BAMBOO BASED ACTIVATED CARBON FOR REMOVAL OF LEAD FROM AQUEOUS SOLUTION

BY

YOSEF ASRAT WAJI

A Thesis Submitted To Collage of Biological and Chemical Engineering Department of Environmental Engineering for Partial Fulfilments Master of Science in Environmental Engineering

ADDIS ABABA SCIENCE AND TECHNOLOGY UNIVERSITY

OCTOBER, 2018

Declaration

I hereby declare that this thesis entitled "***Bamboo Based Activated Carbon for Removal of Lead from Aqueous Solution***" was composed of myself, with the guidance of my advisor, that the work contained herein is my own except where explicitly stated otherwise in the text, and that this work has not been submitted, in whole or in part, for any other degree or professional qualification.

_____

Yosef Asrat

Certificate

This is to certify the thesis prepared by Mr. Yosef Asrat Waji entitled "***Bamboo Based Activated Carbon for Removal of Lead from Aqueous Solution"*** and submitted in fulfillment of the requirements for the Degree of Masters of Science in Environmental Engineering. Complies with the regulation of the University and meets the accepted standards with respect to originality and quality.

_____

Amare Tiruneh (PhD)

Thesis Advisor

Abstract

The presence of heavy metals in surface waters and in wastewater is becoming a severe environmental problem, the objective of the papers were to examine the performance of bamboo activated carbon for adsorption of lead (Pb^{2+}) from aqueous solution. Sorption characteristics of Pb^{2+} through agricultural adsorbent material bamboo based activated carbon was prepared by pyrolysis process with activating agent of potassium hydroxide for adsorption of Pb^{2+} from aqueous solution. The properties of bamboo activated carbon were characterized by scanning electron microscope (SEM), x-ray diffraction (XRD), energy dispersive (functional groups) analysis by FTIR and the physicochemical characteristics of bamboo activated carbon was characterized in terms of the yield rate (40.6gm to 36.0gm), ash (3.5% to 5.53%), porosity (0.704% to 0.6926%), and moisture (7.7% to 6.20%)respectively at different carbonization temperature in the chemical activation process using standard methods of analysis. The iodine number was observed to increase at higher activation temperatures. However, at activation temperature around 600°C and above the iodine number practically decreases. Metal removal was observed as pH increases from 3-6, this might be due to decrease in competition between hydronium ions and metal ions for the surface sites when the pH at 5 it reaches the maximum point 99.8% removal efficiency, The performance of 99.8% removal efficiency in simulated wastewater declined to 60.42% at 500^0C, 56.63% at 600^0C and 45.49% at 700^0C. The results were much lower than the removal efficiency of synthetic wastewater containing individual metal ions. This can be justified to the fact that the presence other organic solvents, heavy metals ions (cadmium, chromium), binders, additives, BOD5, COD, colors and other toxic material in the solution affect the removal efficiency by competing one another to the adsorbent site. The data were analyzed by the two models Langmuir and Freundlich, freundlich isotherm model more favorable than the Langmuir were it got high correlation coefficient (0.9496). Therefore, it is recommended that bamboo stem based activated carbon can be used, as an abundant source for the removal of heavy metal (Pb^{2+}) as an alternative to more costly materials.

***Key words**: Adsorption of lead, bamboo activated carbon, isotherm study*

Acknowledgment

First and for most, I would like to thank the Almighty God for giving me courage through all challenges I faced in completion of this thesis. I express my deep sense of gratitude to my research advisor, Dr. Amare Tiruneh for his assistance, invaluable comments, suggestions and excellent supervision throughout my research work I must also thank him for the appreciation and encouragement showed towards me, which was very helpful and very much precious. Secondly my thanks go to Dr. Getachew who helped me during scanning electron microscope he is humble, patience and kind I would like to thank him very much, finally i would like to thank all my family, and my friends who stood beside me through the difficult times.

Table of Contents

List of Tables

List of Figures

List of Abbreviation

AASTU: Addis Ababa science and Technology University

AC: Activated carbon

ASTM: American Standard Test Method

BAC: Bamboo activated carbon

BOD5: Biological oxygen demand

COD: Chemical oxygen demand

EPS: Extracellular Polymeric Substances

FTIR: Fourier transforms infrared

MC: Moisture content

SEM: Scanning Electron microscope

TMR: Transparency market Research

XRD: X-ray diffraction

1. Introduction

1.1 Background

Heavy metals occur in immobilized form in sediments and as ores in nature. However due to human activities these metals are mobilized and released and natural biogeochemical cycles is disrupted causing increased deposition of heavy metals in terrestrial and aquatic environment (Holant, 1995). Because of their non-biodegradable and persistent nature, heavy metals pollution poses a significant threat to the environment (Mahamood, 2010 and Fu & Wang, 2011). The presence of heavy metals in surface waters and in waste water is becoming a severe environmental problem. Furthermore, because of their non-degradability, they can accumulate in the food chain, there by presenting a significant danger to human health. Lead is one of the major toxic pollutants, which entered the water streams through various industrial operations such as battery manufacturing, metal painting and finishing, paint manufacturing, petroleum refining, glass manufacturing and many others. Environmental and occupational exposure of these heavy metals can cause severe toxic damage to neuronal system, kidneys, reproductive system, liver and brain (Bahadir *et al*. 2007).

The generation of heavy metals from different industrial activities mentioned above, in the environment is of major concern because of their toxicity, bio-accumulating tendency, and threat to human life and the environment through a process of bio magnification; they further accumulate in food chains (Abia, 2006 and Alluri *et al*. 2007).

Since lead do not degrade in the environment, their safe and effective disposal is important. Various treatment options used to remove heavy metals from aqueous solution include chemical precipitation, electrochemical reduction, ion exchange, reverse osmosis, membrane separation and adsorption. Compared with other treatment method, adsorption appears to be an attractive process due to its simplicity, effectiveness and economical in the removal of heavy metals from aqueous solution. Commercial activated carbon is the most widely used adsorbent for removal of

Pollutants from aqueous medium; however its use is restricted due to high cost. Thus, efforts have been geared toward the use of low cost adsorbents for adsorption processes.

According to Babarinde he state that activated carbons can be prepared from a large variety of raw- materials with a high carbon content and low levels of inorganic compounds. While there has been much indigenous effort in converting coal, wood, coconut, bagasse, palm shell and maize Cob into activated carbon, certain initiatives were necessary for an investigative study of conversion of bamboo (Babarinde, 2002).

Adsorption of bamboo charcoal is theoretically classified into physical adsorption and chemical adsorption. Physical adsorption is caused by molecule acting force (van der Waals force) between adsorbent and adsorbate that doesn't change the surface composition of adsorbent and the situation of the molecule of adsorbate. Chemical adsorption is by chemical bond between adsorbent and adsorbate in which the exchange and transference of electrons happen to result in rearrange of atoms and chemical bond formation or destroying.

1.2 Statement of Problem

The increasing use of heavy metals over the past few decades has inevitably led to an increased flux of metallic substances in ground water and surface water, as well as drinking water, and posed serious ecological and health risks, lead ($Pb^{2+)}$ is considered to be an extremely toxic metal without known biological function, toxic when they are not metabolized by the body and accumulate in the soft tissues they can enter the bodies of humans via the food chain (The scientific community has set a level of 10µg/deciliter of blood as a threshold level of concern with respect to lead poisoning. The presence of lead and some other heavy metals as an impurity in paint industry, battery industry glass industries are the most common ones. Removals of heavy are not an easy task it requires advanced technologies and cost.

The demand for activated carbon is increasing owing to the increased utility of the carbon materials in pollution control. As the applications of activated carbon are immense, the gap between demand and supply is ever widening. As a result, cost of activated carbon is increasing, this is due to the use of non-renewable and relatively expensive starting material such as either coal based or petroleum pitch based which are prone to exhaustion and unjustified in pollution control applications (Tan *et al*. 2007). In Ethiopia an estimated one million hectares of natural bamboo forest, the largest in the African continent, the species can be used in wood working and furniture industries substituting wood, for pulp and paper production and for construction purposes.

The large abundance of leftover bamboo from construction, wood work industries the leaf, stem and a root part are able to use in production of activated carbon. In our country Ethiopia exist a light industry like paint; most this industry uses heavy metal for the purpose of increasing the durability and ability of binding on the surface of the wall, the leftover heavy metal are then discharged without being treated these situation gets our surrounding became complicated and leads to sever problem. Locally abundance bamboo stems were used as an alternative mechanism to production of activated carbon for reduction of the lead from the paint industry effluent.

1.3 Objectives

1.3.1 General objective

The general objective of the study is to examine the performance of bamboo based activated carbon for removal of heavy metal (lead) from aqueous solution.

1.3.2 Specific objectives

- Investigation of Physicochemical characteristics of bamboo based activated carbon.
- Seeing the effects under various experimental conditions of *pH, concentration of heavy metal ion, adsorbent dosages* on adsorption behavior of heavy metal (lead).
- Evaluation of the optimum condition for the removal of lead from the real wastewater (Nefas silk paint wastewater) stream.
- Analyze the data obtained from the adsorption experiment using the sorption isotherm models homogenous and heterogeneous surface nature of BAC (Freundlich and Langmuir).

1.4 Significance of the Study

A major challenge of many industries is finding solutions that equate to positive environmental and economic impacts regarding the treatment of their effluents. Solution to this challenge is of uttermost importance to enable improvement of water quality. Since the activated carbons employed in this study are derived from natural sources (bamboo stem), they can represent economic and environmental friendly alternatives to conventional adsorbent (commercial activated carbon).

1.5 Scope of the study

The scope of the study were mainly sticks with the adsorption performance of bamboo based activated in aqueous solution at a laboratory scale at different experimental parameters (*pH, metal ion concentration and adsorbent dose).*

2. Literature review

Heavy metals exist in rocks, as ores, such as sulphides (iron, arsenic, lead, lead, zinc, cobalt, gold-silver and nickel sulphides), oxides (aluminum, manganese, gold, selenium and antimony) and both sulphides and oxides from which they are recovered as minerals (Duruibe, 2007). This anthropogenic activity in recovery of minerals causes drastic change in geological and biochemical balance (Sudhakar *et al*. 2012).

Electroplating process, such as, electro less depositions, conversion coating, anodizing cleaning, milling, and etching generate heavy metals (such as cadmium, zinc, lead, chromium, nickel, copper, vanadium, platinum, silver, and titanium). Printed Circuit Board (PCB) process generates tin, lead, and nickel solder plates are the most widely used resistant over plates. Others sources for the metal wastes include; the wood processing industry where a chromatid copper-arsenate wood treatment produces arsenic containing wastes; inorganic pigment manufacturing producing pigments that contain chromium compounds and cadmium sulfide; petroleum refining which generates conversion catalysts contaminated with nickel, vanadium, and chromium; and photographic operations producing film with high concentrations of silver and Ferro cyanide (Barakat, 2011). The food-chain pyramid receives metals through anthropogenic causes and on top of the pyramid, man receives pre-concentrated metal toxicity. There are also cases of no anthropogenic pollution. As the need for fresh sources of drinking water is becoming urgent worldwide, sustainability in the field of water separation processes and in general, its significance for the chemical and process industry has been noted.

The presence of copper, zinc, cadmium, lead, mercury, iron, nickel and others metals, has a potentially damaging effect on human physiology and other biological systems when the tolerance levels are exceeded (Demirbas, 2008). Hence, there is the need for proper understanding of the conditions, such as the concentrations and oxidation states, which make them harmful, and how biotoxicity occurs.

Exposure to some metals, such as mercury and lead, may also cause development of autoimmunity, in which a person’s immune system attacks its own cells. This can led to joint

diseases such as rheumatoid arthritis, and diseases of the kidneys, circulatory system, nervous system, and damaging of the fetal brain (Barakat, 2011). The poisoning effects of heavy metals are due to their interference with the normal body biochemistry in the normal metabolic processes.

2.1 Advanced removal methods of heavy metals

Methods for removing metal ions from aqueous solution mainly consist of physical, chemical and biological technologies. Conventional technologies, such as chemical precipitation, lime coagulation, solvent extraction, membrane filtration, reverse osmosis, ion exchange and adsorption, are being used for the removal of heavy metal ions from aqueous wastes (Holan & Volesky, 1995). The process description of each method is presented below (Krzywonos, 2014).

2.1.1 Reverse osmosis

The reverse osmosis process depends upon a semi-permeable membrane through which pressurized water is forced. Reverse osmosis, simply stated, is the opposite of the Natural osmosis process of water. Osmosis is the name for the tendency of water to migrate from a weaker saline solution to a stronger saline solution, gradually equalizing the saline composition of each solution when a semi-permeable membrane separates the two solutions. In reverse osmosis, water is forced to move from a stronger saline Solution to a weaker solution, again through a semi-permeable membrane. Because molecules of salt are physically larger than water molecules, the membrane blocks the Passage of salt particles (Barakat, 2011). The end result is desalinated water on one side of the membrane and a highly concentrated, saline solution of water on the other side. The disadvantage of this method is that it is expensive (Ahalya *et al.* 2003).

2.1.2 Electrolysis

In this process, the ionic components (heavy metals) are separated through the use of semipermeable ion selective membranes. Application of an electrical potential between the two electrodes causes a migration of cations and anions towards respective electrodes. Because of the alternate spacing of cation and anion permeable membranes, cells of concentrated and dilute salts are formed. The disadvantage is the formation of metal hydroxides, which clog the membrane.

2.1.3 Ultra-filtration

They are pressure driven membrane operations that use porous membranes for the removal of heavy metals. The main disadvantage of this process is the generation of sludge.

2.1.4 Ion exchange

An ion exchanger is a solid capable of exchanging either cations or anions from the surrounding materials held by electrostatic forces on the exchange resin. Commonly used matrices for ion exchange are synthetic organic ion exchange resins. The disadvantages include; high cost, partial removal of certain ions, and it cannot handle concentrated metal solution as the matrix gets easily fouled by organics and other solids in the wastewater.

2.1.5 Chemical precipitation

Precipitation of metals is achieved by the addition of coagulants such as alum, lime, iron salts and other organic polymers. The large amount of sludge containing toxic compounds produced during the process is the main disadvantage.

2.1.6 Phytoremediation

Phytoremediation is the use of certain plants to clean up soil, sediment, and water contaminated with toxic metals. The disadvantages include that it takes a long time for removal of metals and the regeneration of the plant for further biosorption is difficult.

Due to the disadvantages mentioned above, like incomplete metal removal, high reagent and energy requirements, generation of toxic sludge or other waste products that require cost effective alternative technologies or adsorbents for the treatment of metal containing wastewaters are needed. Natural materials that are available in large quantities, or certain waste products from agricultural operations, may have potential as inexpensive adsorbents. Due to their low cost, after these materials have been expended, they can be disposed of without regeneration. (Kadirvelu *et al*. 2001 and Kanamadi, 2003).

2.2 Adsorbent development

The most widely used carbonaceous materials for the industrial production of activated carbons are coal, wood and coconut shell. These types of precursors are quite expensive and often imported. This makes it necessary, for developing countries, to find a cheap and available

feedstock for the preparation of activated carbon for use in industry, drinking water purification and waste water treatment. The term "biomass", according to EU Directive 2003/30/EC, refers to the biodegradable fraction of products, wastes, and residues from agriculture (including vegetable and animal substances), forestry, and related industries, as well as the biodegradable fraction of industrial and municipal waste.

Moreover, if 42.14% of the world market volume in 2012 was directed for water treatment and for 35.21% air purification, there is a tendency for the activated carbon market to advance towards the food, pharmaceutical and medical industry (TMR, 2016). AC is a highly porous carbonaceous material, with an elevated internal surface area and functional groups in its surface with an adsorption affinity for various contaminants. AC is obtained from controlled pyrolysis of a raw material, where temperatures vary from 400 to 1000°C, and consequent physical and chemical activation.

There are two ways of using activated carbon: liquid and gas. The most known applications are water treatment (remedying bodies of water, effluent treatment and sanitary landfill and water filters lixiviate); air purification (toxic gases and combustion gases removal); food and drink industrialization (sweetener purification, edible oils, glycerin, wine, organic and inorganic acids); pharmaceutical production (color and compounds removal from some drugs); medicinal use (toxic products elimination; tablets, curatives, odor control filters and masks productions), use in automotive vehicles (adsorbing combustible emissions or inside odors from them). It also is useful on oil refineries as a support for catalysts, gas storage, dehumidification (mold elimination and food conservation); in mining; and in the electronic industry (Bautista-Toledo *et al*. 2014).

The adsorption process is being widely used by various researchers for the removal of heavy metals from waste streams and activated carbon has been frequently used as an adsorbent (Khan *et al*. 2004). Activated carbon is used as adsorbent material but it is expensive, therefore, research interest into production of cheaper adsorbents. Recently, apart from activated carbon various low cost adsorbents, derived from agricultural waste, industrial byproduct, natural material, or modified biopolymers, have been developed and applied for the removal of heavy metals from metal contaminated wastewater (Barakat, 2011).It was said that it is particularly interesting to develop an adsorbent demonstrating a high adsorption capacity and low cost for

removing various pollutants from contaminated waters; inorganic chemical adsorbents have thus been studied widely, aiming at providing an alternative to activated carbon in the treatment of surface and ground water and industrial effluents (Jiang, 2012).

Activated carbons, on the other hand, are among the effective adsorbents because of their excellent sorption capacity. Their use has been often prohibited mainly due to their relatively high cost, low selectivity and regeneration problems; they can be regenerated by thermal desorption of the adsorbed substances or by liquid phase extraction in the case of soluble adsorbed species, although the former method is not very environmental friendly and leds to partial mass loss of the adsorbent (Chen *et al.* 2007).

The microporous nature of the majority of porous activated carbons is well suited to many applications, including molecular sieving, sorption and catalytic reactions of small molecules. Of course, there are numerous other potential applications, such as sorption of dyes, in which the presence of wider pores, preferably in the mesoporous range, would be advantageous (Asouhidou *et al.* 2009).

Metal-loaded mesoporous carbons have been also investigated as efficient adsorbents for organic molecules due to the high mesopore volume as well as to the specific interactions with the supported metals. Therefore, there has recently been a considerable interest in the synthesis of (mesoporous) carbons with high surface areas and tailored pore structures. Specifically, heavy metals are discharged by industries such as agrochemical, petrochemical and fertilizers, whereas dyes are found principally in effluents of dye manufacturing industries, electroplating factories, distilleries and food companies (Kasnejad *et al.* 2012). It was further reported that an anionic dye favored the electrostatic interactions with heavy metals or might create new specific sites for the adsorption process (Tovar-Gomez R. *et al*. 2012).

Another well-known metals separation application of activated carbon, apart from organics, is certainly that in gold processing, which however does not constitute a typical case of pollutant removal. Activated carbon, usually in granulated form, has been used to preferentially adsorb gold and silver occurring as cyanide complexes. Powdered activated carbon that offers more

surface area for adsorption and is less costly to manufacture, has limitations and difficulties are encountered in its separation; the application of flotation downstream was suggested as an effective separation technique of the loaded particles.

A wide range of physical and chemical processes are available for the removal of pollutants from effluents. A major drawback with those treatment systems is sludge production, and, high operational cost and some of them are complicated for management. This actually makes the application of these technologies to be limited only in developed countries. In response to this challenge a different attempt were undertaken to produce a media which was feasible and cost effective to use by the majority (Alebel .A, 2010).

Plant biomass is a natural renewable resource that can be converted into useful materials and energy. The idea of using bamboo to produce activated carbon rises in this context. Bamboo is a grass, the most diverse group of plants in the grass family. With this regard, the prevalence of adsorption separation in the environmental chemistry remains an aesthetic attention and consideration abroad the nations, owning to simplicity of design, ease of operation, insensitivity to toxic substances and complete removal of pollutants even from dilute solutions (Foo and Hameed, 2010).

Despite its prolific use in adsorption processes, the biggest barrier of its application by the industries is the cost-prohibitive adsorbent and difficulties associated with regeneration (Foo and Hameed, 2009). Realizing the complication, a growing exploitation to evaluate the feasibility and suitability of natural, renewable and low-cost materials as alternative adsorbents has been exerted. With this regard, activated carbon is the most promising adsorbent in environmental application.

2.3 Activated Carbon

Activated carbon is broadly defined to include a wide range of amorphous carbon based materials prepared in such a way that they exhibit a high degree of porosity and an extended surface area and different surface functional groups; including carboxyl, carbonyl, phenol, quinone, lactone and other groups bound to the edges of the graphite-like layers. Both surface

chemistry and texture of carbons are affected by the nitrogen source and the type of oxygen functionalities pre-existing on the surface (Deliyanni, 2011).

They are therefore regarded as good adsorbents for the removal of heavy metal ions and other inorganic substances, as well as many organic compounds from liquid and gas phases. (Bansal and Goyal, 2005). Moreover, all non-carbon impurities are removed and the surface is oxidized. Activated carbons are used in a number of industrial applications including separation and purification technologies, catalytic processes, biomedical engineering, and energy storage, among others. Activated carbon is one of the most commonly used adsorbents in the removal process of industrial pollutants, organic compounds, heavy metals, herbicides, and dyes, among many others toxic and hazardous compounds (Peláez-Cid and Teutli-León, 2012).

The extensive application of activated carbon is mainly due to its relatively low cost with respect to other adsorbents, wide availability, high performance in adsorption processes, surface reactivity and the versatility to modify its physical and chemical properties for synthesizing adsorbents with very specific characteristics (Haro *et al*. 2011).Activated carbon adsorption is most commonly applied in industrial wastewater treatment to meet stringent regulations for discharge into receiving waters. In industrial wastewater treatment, activated carbon adsorption can be utilized as a separate unit process. It may be placed after various physicochemical treatment steps such as coagulation/clarification, filtration, and dissolved air flotation. Another alternative is to use activated carbon adsorption prior to biological treatment to remove compounds which might be toxic to a biological system. However, the most widely adopted procedure is to place activated carbon adsorption as a tertiary or advanced treatment step subsequent to biological treatment for removal of refractory organics. To some extent this procedure may also be effective in the removal of inorganics (Cecen, 2011).

Most of the commercial activated carbons are either coal based or petroleum pitch based which are prone to exhaustion. Their global distribution is non-uniform. As the applications of activated carbon are immense, the gap between demand and supply is ever widening. This may in due course result in scarcity of the material in addition to becoming expensive. This situation necessitates the need for the exploration of new sources of carbon materials with desired physico chemical properties namely, high specific surface area, micro or mesoporosity or both,

depending on the end application, surface functionality, thermal stability, carbon purity, adsorptive capacity and chemical composition.

Traditionally, the activated carbons used in wastewater treatment are obtained from coal/lignite, wood or animal bones but, recently, there is a growing interest in the use of alternative and low-cost precursors for their production (Elizalde-González and Hernández-Montoya, 2007, Altenor *et al*. 2009, Mohamed *et al*. 2010).The production of activated carbons from agricultural and industrial wastes is usually justified by two factors: the unique properties of these precursors and the possibility of mass production at an affordable cost. Lignocellulosic materials have been and will be with mankind forever and they hold a promise of renewable and inexhaustible supply of carbon materials provided suitable methods of production are developed. In addition they are more evenly distributed throughout the globe relative to either coal or petroleum (Durán-Valle, 2012). Thus the lignocellulosic material, a regenerable natural resource, is a viable option for the generation of carbon materials rather than fossil fuels. Preparation of Activated Carbon, According to (Hernández-Montoya *et al.* 2012), the preparation of ACs from lignocellulosic materials involved two processes, the carbonization and the activation, which can be performed in one or two steps depending on the activation method (physical or chemical, respectively). The carbonization consists of a thermal decomposition of raw materials, eliminating non-carbon species and producing a fixed carbon mass with a rudimentary pore structure (very small and closed pores are created during this step). On the other hand, the purpose of activation is to enlarge the diameters of the small pores and to create new pores.

2.3.1 Activated Carbon Application as Low-Cost Adsorbent

Nowadays, activated carbon finds wide application in many areas, but especially in the environmental field. Aside from environmental pollution control, activated carbon is mainly used in industry in various liquid and gas phase adsorptions. Among liquid phase applications one can list food processing, preparation of alcoholic beverages, decolorization of oils and fats, product purification in sugar refining, purification of chemicals (acids, amines, gylcerin, glycol, etc.), enzyme purification, decaffeination of coffee, gold recovery, refining of liquid fuels, purification in electroplating operations, purification in the clothing, textile, personal care, cosmetics, and pharmaceutical industries, and applications in the chemical and petrochemical industries.

Gas phase applications include recovery of organic solvents, removal of sulfur-containing toxic components from exhaust gases and recovery of sulfur, biogas purification, and use in gas masks, among others. Activated carbon is also used in medical and veterinary applications, soil improvement, removal of pesticide residues, and nuclear and vacuum technologies (Cecen, 2011).

Activated carbon adsorption is most commonly applied in industrial wastewater treatment to meet stringent regulations for discharge into receiving waters. In industrial wastewater treatment, activated carbon adsorption can be utilized as a separate unit process. It may be placed after various physicochemical treatment steps such as coagulation/clarification, filtration, and dissolved air flotation. Another alternative is to use activated carbon adsorption prior to biological treatment to remove compounds which might be toxic to a biological system. However, the most widely adopted procedure is to place activated carbon adsorption as a tertiary or advanced treatment step subsequent to biological treatment for removal of refractory organics.

Nowadays, activated carbon finds wide application in the treatment of wastewaters generated from industries such as food, textile, chemical, pharmaceutical, pesticides and herbicides production, coke plant, munitions factories, petroleum refineries and storage installations, organic pigments and dyes, mineral processing plants, insecticides, pesticides, resins, detergents, explosives, and dyestuffs. It is also employed in the treatment of sanitary and hazardous landfill leachates (Cecen, 2011).

2.3.2 Parameters for Activated Carbon Preparation

Research has shown that carbon properties such as specific surface area, porosity, density and mechanical resistance depend greatly on the raw material used. However, it may be possible to modify these parameters changing the conditions in the pyrolysis process of the agricultural and industrial waste (lignocellulosic) materials. The pore size distribution and surface area are determined by the ratio between the mass of the chemical agent and the raw material. Besides, activation time, carbonization temperature and heating rate are important preparation variables for obtaining ACs with specific characteristics (Mohamed *et al*. 2010).

2.3.4 Activating Agent

A multitude of activating agents has been extensively employed for the production of activated carbon materials with desired pore structure. The purpose of activation is to create and develop (volume and size) porosity in the carbon material and thereby increase the adsorptive capacity. In preparation of activated carbon, the lignocellulosic precursor is treated primarily with a chemical agent, such as H_3PO_4, H_2SO_4, HNO_3, NaOH, KOH or $ZnCl_2$ by impregnation or physical mixture and the resulting precursor is carbonized at temperatures between 400 and 800ºC under a controlled atmosphere.

2.3.5 Carbonizing Temperature

It has the most influence over the activated carbon's quality during the activating process. It must be at least 400 °C to ensure the complete transformation of organic compounds (present in agricultural and industrial waste (lignocellulosic) precursors) into graphene structures. The degree of specific surface area development and porosity is incremented on par with the carbonizing temperature (Olivares-Marín *et al*. 2006).

2.3.6 Carbonizing Time

This parameter must be optimized to obtain the maximum porosity development while still minimizing the material's loss due to an excessive combustion.(Bouchelta *et al*. 2008) have shown that the yield percentage decreases with increase of activation temperature and hold time. Carbonization times ranging from 1 hr (Rajeshwarisivaraj *et al*. 2001, Wu *et al*. 1999) up to 14 hr (Rajeshwarisivaraj *et al*. 2001) have been used in charcoal production.

2.3.7 Mass Ratio of Precursor and Activating Agent

The complete saturation of agricultural and industrial waste (lignocellulosic) precursor must be ensured to develop the adsorbent porosity with the minimum activating agent consumption. This leads a minor consumption of chemical compounds and a better elimination of the excess during the carbon washing process. The effect of the increase in proportion of the impregnation over the carbon porous structure is greater than the one obtained with the increase of carbonizing temperature (Olivares-Marín *et al*. 2006a).

2.3.8 Heating Speed

Regularly, heating ramps (the smart control and monitoring ensure the system is energy efficiently and provide good operational conditions) with a low speed are used for preparation of activated carbon. This approach allows the complete combustion of material precursor and favors a better porosity development. Rapid heating during pyrolysis produces macroporous residue (Heschel and Klose, 1995).

2.4 Characteristics and Technique of Characterization

Properties such as specific surface area, pore size, and pore size distribution are important material characteristics not only for AC but also in many processing applications, including those for catalysts, sintered materials, pharmaceutical products, and chromatographic carriers.

Gas adsorption measurements are widely used for the characterization of a variety of porous solids. Of particular importance is the application of physisorption (physical adsorption) for the determination of the surface area and pore size distribution. Nitrogen (at 77 K) is the recommended adsorptive for determining the surface area and mesopore size distribution, but it is necessary to employ a range of probe molecules to obtain a reliable assessment of the micropore size distribution. An alternative technique to gas adsorption is mercury porosimetry used for macropore size analysis. For operational reasons, krypton adsorption (at 77 K) is usually adopted for the determination of relatively low specific surface areas ($<2 m^2 g^{-1}$), but this technique cannot be employed for the study of porosity (Durán-Valle, 2012).

The iodine number is the amount of iodine, in milligrams, adsorbed per gram of carbon when the equilibrium concentration (Ce) of iodine is 0.02 M. It is the most fundamental parameter used to characterize activated carbon performance. It is a measure of activity level (higher number indicates higher degree of activation), often reported in mg/g (typical range 500–1200 mg/g). It is a measure of the micropore content of the activated carbon (0 to 20 Å, or up to 2 nm) by adsorption of iodine from solution. It is equivalent to surface area of carbon between 900 m^2/g and 1100 m^2/g (Baseri *et al*. 2012, Sivakumar *et al*. 2012) the micropores are responsible for the large surface area of activated carbon particles and are created during the activation process. It is in the micropores that adsorption largely takes place (Ekpete and Horsfall, 2011).

It has been established that the iodine number in mg/g gives an estimate of the surface area in m^2/g (Gergova *et al*. 1994) and measures the porosity for pores with dimensions between 1.0 - 1.5nm (Collin *et al*. 2006). The removal of iodine by the activated carbons is related to their porosity characteristics which determine the degree of accessibility of these molecules. A lower iodine number can be ascribed to the presence of pores narrower than 1.0 nm, which make up most of the structure of these carbons (Khadija *et al*. 2008). For carbons prepared by physical activation, the mass of iodine absorbed was generally low (92 to 261 mg/g), whereas carbons prepared by chemical activation exhibited a higher iodine adsorption capacity (240-268 mg/g) indicating improved porosity. These carbons contain mostly micro pores with a small contribution of mesoporosity. AC recommended for water treatment, are to show iodine values ranging from 600 to 1100 mg/g (AWWA, 1991).

2.5 Biosorption

Biosorption, a biological method of environmental control can be an alternative to Conventional waste-treatment facilities. Several suitable agricultural by-products lignocellulosics like bamboo materials (*Lo S.. et.al* 2012). Have been investigated in the recent years as activated carbon precursors and are still receiving attention. In recent years, there has been a rising expansion of the activated carbon (AC) global market. According to the Transparency Market Research (TMR), the product transactions reached, in 2012, 1.913 billion dollars and the predictions are that at the end of 2019 the numbers surpass the mark of 4.180 billion, presenting an annual increase rate of 11.9% in the 2013-2019 period.

Metals can be removed and concentrated from solutions by using biomass material whereby the metal is adsorbed by chemical sites naturally present and functional dead, inactive or plant based biomass (Volesky, 2001 and Sulaiman, 2015). A vast array of biological materials, especially bacteria, algae, yeasts and fungi have received increasing attention for heavy metal removal and recovery due to their good performance, low cost and large available quantities. The biosorbent, unlike mono functional ion exchange resins, contains variety of functional sites including carboxyl, imidazole, sulphydryl, amino, phosphate, sulfate, thioether, phenol, carbonyl, amide and hydroxyl moieties (Wang & Chen, 2009).

According to different researchers on this area, their work revealed that dead cells have shown ability to bind metal ions through various physico-chemical mechanisms this resulted in an increase in the number of new potential applications (Zabochnicka *et al.* 2014) The advantage of biosorption is in using biomass raw materials which are either abundant seaweeds or wastes from other industrial operations fermentation wastes (Volesky, 2001).

Biosorption have several applications in different industries especially the heavy metals processing ones, which include metal plating, metal processing, mining, battery manufacturing and metal finishing operations (Sulaiman, 2015). Applying biotechnology in controlling and removing metal pollution has been paid much attention, and gradually becomes hot topic in the field of metal pollution control because of its potential application (Holan & Volesky, 1995).

Generally, metal ions removal by biosorption process is more advantageous than conventional treatment methods because of the following reasons (Khan *et al*. 2004, Kanamadi, 2003 and Ahluwalia & Goyal, 2007); Low investment and low operational cost, it requires little processing, it is abundant in nature, it is a byproduct or waste material from another industry High removal efficiency, reducing residual metals to below 1 ppb in many cases, minimization of chemical and/or biological sludge, no additional nutrients requirement that cause disposal and space problems, regeneration of biosorbent and Possibility of metal recovery.

The regeneration of biosorbent is important part of biosorption process that decrease the cost of biosorbent utilized for further treatment and in opening the possibility of recovering the metals extracted from the liquid phase. For this purpose, it is desirable to desorb the sorbed metals and to regenerate the biosorbent material for another cycle of applications. The desorption process should yield the metals in concentrated form. And it should restore the biosorbent close to the original state for effective reuse with undiminished metal uptake and no physical change or damage to the biomass. Dilute mineral acids have been used for removal of metal from biomass (Mahamood, 2010).

While the regeneration of the biosorbent may be accomplished by washing the metal-laden one with an appropriate solution, the type and strength of this solution would depend on just how the

deposited metal has been bound. In batch tests of desorbing solutions, one has to realize that the desorbed sorbate metal ions stay in the solution and a new equilibrium is established between that and the one remaining (Volesky, 2001). Biosorption have several applications in different industries especially the heavy metals processing ones, which include metal plating, metal processing, mining, battery manufacturing and metal finishing operations (Sulaiman, 2015). Applying biotechnology in controlling and removing metal pollution has been paid much attention, and gradually becomes hot topic in the field of metal pollution control because of its potential application (Holan & Volesky, 1995).

2.5.1 Agricultural wastes and cellulose based materials

They are composed of lignin and cellulose, as well as other polar functional groups containing compounds, which include alcohols, aldehydes, ketones, carboxylates, phenols and ethers which have an ability to bind metal ions from solutions (Khan *et al*. 2004 and Lesmana *et al*. 2009).
Agricultural byproducts such as rice husk (Ajmal *et al*. 2003, Wong *et al*. 2003 and Sobhanardakani *et al*. 2013), tea waste (Thakur & Parmar, 2013 and Dwivedi, 2014), ground nut shell (Prabha & Udayashankara, 2014), lemon and peel (Husoon, 2013), moringaoleifera (Ravikumar, 2013 and Mataka *et al*. 2010), neem leaf powder (Venkateswarlu *et. al*, 2007 and Sharma & Bhattacharyya, 2005), coconut shell (Okafor *et al*. 2012), saw dust (Yu *et al*. 2001) etc. are some typical examples investigated as metal ions adsorbent by researchers.

2.6 Biosorption metal uptake mechanism

Technically biosorption process involves a solid biosorbent phase and a liquid phase; often water which contains a dissolved species to be sorbed (sorbate, a metal ion) till equilibrium is established (Alluri *et al*. 2007 and Mosbah & Sahmoune, 2013). Due to higher affinity of the sorbent for the sorbate species the latter is attracted and bound with different mechanisms such as ion exchange, complexation, precipitation, chelation, coordination, reduction and physical adsorption (Tsezos et al., and Kanamadi, 2003).

Generally, biosorption mechanisms can be classified based on various criteria (Alluri *et al*., 2007 and Kanamadi, 2003). On the basis of cell's metabolism, biosorption mechanisms can be metabolism dependent and non-metabolism dependent. On the other hand, according to the

location of sorbet species biosorption can be classified as extracellular accumulation/ precipitation, cell surface sorption/precipitation and intracellular accumulation.

Cell membranes play an important role in the process of sorption because of the availability of various functional groups such as carboxylic, hydroxyl phosphate amine which can be considered as a complex ion exchanger similar to commercially available resins (Zabochnicka *et al.* 2014). Metal ions are adsorbed and transported across the membrane in the same mechanism by which metabolically important ions such as potassium, magnesium, and sodium are conveyed (Alluri *et al*. 2007). The term adsorption can be used as a general term and includes several passive, i.e. non-metabolic, mechanisms such as: complexation; chelation; co-ordination; ion exchange; precipitation; reduction (Tsezos *et al*. 2007).

2.6.1 Complexation

Complex formation of metal ions with organic molecules involves ligand centers in the organic species i.e. the presence of an atom or atoms having lone pair electrons to donate. Complexation may be electrostatic or covalent and the simplest case is complexation by a mono-dentate ligand such as RNH_2. To approach and elucidate biosorption mechanisms, a significant part of the recent advances in biosorption are based on the classification of elements according to the hard-soft acid-base classification (Pearson's classification). "Hard acids", metals such as Na, K, Ca, Mg, often essential nutrients for microbial growth, bind preferentially to oxygen containing "hard bases", ligands such as OH^-, $HPO4^{2}$-, $CO3^{2-,}$ $R\text{-}COO^-$, and =C=O. Soft acids, metals such as the precious metals Ag, Au, Pt, Pd are bound covalently to the cell wall by "soft bases", ligands containing nitrogen or sulfur.

2.6.2 Chelation

Organic molecules containing more than one functional group with donor electron pairs can simultaneously donate these to a metal atom. This can result in the formation of a ring structure involving the metal atom a process termed 'chelation'. Chelated compounds are more stable than complexes involving mono-dentate ligands. Stability tends to increase with the number of chelating sites available on the ligand.

2.6.3 Coordination

Metal atoms have preferences for specific donor atoms ("hard/hard" / "soft/soft") and the stereo chemical arrangements that play an important role in the binding with the available ligands on the microbial cell.

2.6.4 Anion Exchange

Anion exchange on biopolymers can take place on a variety of organic nitrogen based groupings. In proteins, amino (lysyl side chain and N-terminal) imidazole (histidyl) and guanidine (arginyl) groupings are common centers of positive charge. Centers of positive charge in nucleic acids will occur with protonation of amino groups on purine or pyrimidine rings or with protonation of heterocyclic nitrogen atoms. Polysaccharides as a group are acidic or neutral macromolecules with basic functional groups being rare and arising as unacetylated amino sugars.

2.6.5 Cation Exchange

In biopolymers the most likely cation binding invisible groups to be involved are: carboxyl, organic phosphate and organic sulfate. Carboxylic acids are widely distributed in biopolymers being most commonly found as side-chain constituents of proteins, the uranic, neuraminic and muramic acids, and related substituted monosaccharides of polysaccharides. Phosphodiester links impart negative charge to the nucleic acid backbone while both diester and monoester groupings are found most commonly in bacterial polysaccharides and related macromolecules. Lipoprotein and lipopolysaccharides are also likely to contain phosphodiester as part of the lipid moiety.

Sulphate esterified to carbohydrate hydroxyl groups is common in connective tissue and algal polysaccharides and is for most purposes likely to provide the greatest negative charge density, at very low pKa, among the charged biopolymers. Ester sulfate and phosphomonoester groupings also occur in proteins. Hydroxyl groups of serine, threonine and tyrosine are available for specific translational modifications. Phenolic hydroxyl is also capable of providing weak negative charge and binding potential.

2.6.6 Precipitation

Metal precipitation is also involved in biosorption process. The precipitates may be formed and remain in contact with or inside the microbial cells or may be independent of the solid phase of

the microbial cell. In the latter case, the presence of the solid phase-microbial cell or biofilm also plays a favorable role in the phenomenon of precipitation. Metal precipitates may also be formed.

This may be more easily understood when metals are bound to Extracellular Polymeric Substances (EPS) excreted by some prokaryotic (bacteria, archaea) and eukaryotic (algae, fungi) microorganisms. Purified products from isolated cells such as glucan, mannan, and chitin accumulate greater quantities of cations than the intact cells, proving that biomolecules can form metal precipitates. Further, it may be dependent on the cell's metabolism if, in the presence of toxic metals, the microorganism produces compounds that favour the precipitation process. Precipitation may not be dependent on the cells' metabolism, if it occurs after a chemical interaction between the metal and cell surface.

2.7 Factors affecting biosorption

The most important factors that should be taken into account when considering biosorption are: (i) the type and nature of the biomass; (ii) initial solute concentration; (iii) biomass concentration (biosorbent dosage/solution volume) in solution; (iv) physicochemical factors like temperature, pH, and ionic strength (Chimie, 2014). Apart from the physico-chemical factors such as pH, the presence of other anions and cations, metal speciation, pollutant solubility and form, etc. may also have an influence. With living cell systems, the provision of nutrients and optimal growth conditions is an obvious requirement (Gadd, 2009).

2.7.1 Effect of solution pH

The pH of the metal ion solution is an important parameter for adsorption of metal ions because it affects the solubility of the metal ions, concentration of the counter ions on the functional groups of the adsorbent and the degree of ionization of the adsorbate (Ii et al., 2011). Therefore, pH of solution influences the nature of biomass binding sites and metal solubility; Metal biosorption has frequently been shown to be strongly pH dependent in almost all systems examined, including bacteria, cyanobacteria, algae, and fungi (Holan & Volesky, 1995).

It has been generally reported that in highly acidic medium (pH≈2) the removal of metal ions is almost negligible and it increases by increasing the solution pH up to a certain limit (Abdelghani & Elchaghaby, 2014). This can be explained that decreasing of biosorption levels by lowering

pH can be due to competition between protons and metal ions for the capturing same sites (Igwe, 2006). On the other hand, too high pH value can cause precipitation of metal complexes, so it should be avoided during experiments. For different biosorption system of metal ions, the optimal pH is different. (Wang & Chen, 2006).

2.7.2 Effect of temperature

Temperature influence has more effect in a situation where by metal uptake increases within a temperature range of about 20-30°C, but decreases with an increase of temperature above a critical value (Sulaiman, 2015). Increase in temperature probably weakens the bond formed between the metal ions and the adsorption sites on the adsorbent thereby resulting in an increase in the amount of metal ions adsorbed on the adsorbent. At high temperature, adsorption is expected to decrease due to the increased tendency of the metal ion to escape from the surface of the adsorbent to the solution phase; hence there will bind to be weak adsorption interactions between the adsorbent and the adsorbate (Ojedokun & Solomon, 2016).

2.7.3 Initial concentration

It is generally agreed that the biosorption capacity increases as the initial metal ion concentration in the solution increases, whereas the metal removal percentage (also called removal efficiency) decreases by increasing the metal ion initial concentration (Abdelghani & Elchaghaby, 2014). As a rule, increasing the initial metal concentration results in an increase in the biosorption capacity because it provides a driving force to overcome mass transfer resistance between the biosorbent and biosorption medium (Pahlavanzadeh *et al.* 2010).

2.7.4 Biosorbent dosage

The amount of biomass added in the solution during sorption process also affects the specific metal uptake. In principle, with more biosorbent present, the available adsorption sites or functional groups also increase (Mosbah & Sahmoune, 2013). At low biomass dosage, the number of ions adsorbed per unit adsorbent weight is high. Adsorption capacity is reduced when the biomass dosage increases as a result of lower adsorbate to binding site ratio where the ions are distributed onto larger amount of biomass binding sites (Ojedokun & Solomon, 2016).

2.7.5 Other factors

Biosorption is mainly used to treat wastewater where more than one type of metal ions would be present; the removal of one metal ion may be influenced by the presence of other metal ions (Kanamadi, 2003). Wastewaters are contaminated with various contaminants, including different kinds of metals at the same time, which has an effect on biosorption dynamics. The presence of other substances dissolved in a solute on can inhibit biosorption of metals. This is due to competitiveness between ions of metals that are removed and other ions for binding spots on the surface of cells (Zabochnicka Świątek, 2014).

The age of biomass can influence the properties of cell walls that are crucial for adsorption of heavy metals. According to numerous observations the relationship between the age of biomass and adsorption of heavy metals is not fully understood (Zabochnicka Świątek, 2014). Growth and nutrition of the biomass, and age can also influence biosorption due to changes in cell size, wall composition, extracellular product formation, etc. The surface area to volume ratio may be important for individual cells or particles, as well as the available surface area of immobilized biofilms. The type and nature of the biomass or derived product can be very important including the nature of its application as, e.g. freely-suspended cells or biomass, immobilized preparations, living biofilms, etc. (Gadd, 2009).

3. Materials and Methods

Glass wares (Conical flasks, Pipette, Measuring cylinders, Beakers, Petri plates, filter paper and Test tubes, Desiccators, hot plate was used for preparation of stock solution, transfer of solution, for cleaning and some other purpose throughout the experiment. The instruments and apparatus used throughout the experiment are listed below.

3.1.1 Atomic adsorption spectrophotometer (AAS):

(Microwave Plasma Atomic Emission Spectrometry *Model Agilent 4200 MP-AES)* were used to measure the residual metal ion concentration after filtration by whatman paper of simulated wastewater.

3.1.2 Scanning electron microscope (SEM):

Were used to capture the morphological structure of bamboo activated carbon (*model* INSPECT F 50) at central laboratory of Addis Ababa science and technology university (AASTU) the samples were prepared in carbon black stack material on samples holders after that nitrogen inert gas was applied to removes some unbound BAC from the black carbon stack surface of samples holder, when the energy (electron) became develop the pointer starts to captures at different magnification and resolution of the surface texture.

3.1.3 FTIR instrument:

(*Model* IRAffinity-1S) were used to determine the various functional groups on the surface of the bamboo activated carbon at Leather industry development institute laboratory. Bamboo activated carbon 2mg of bamboo activated carbon powdered mix with 300mg of anhydrous KBr (potassium bromide) the mixture was pressed under vacuum (hydraulic press Model-15).

3.1.4 Orbital shaker:

(Model SSL1) used to mix the simulated contaminant and locally produced activated carbon (BAC) at 200rpm with 120min.

3.1.5 XRD:

(*Model miniflex 600 PxRD)* Used to Investigation of bamboo activated carbon weather crystalline or amorphous nature at Addis Ababa University Chemistry Department.

3.2 Methods

3.2.1 Adsorbent collection and preparation

Ethiopia one of the most endowed countries in bamboo resource in Africa (kassahun, 2003). The two indigenous bamboo species in the country are Africa alpine bamboo (*Arundinaria alphina*) and a monotypic genus of lowland bamboo (*oxytenathera abyssinica*) (Azene, 2007). The species can be used in wood working and furniture industries substituting wood, for pulp and paper production and for construction purposes. The leftover bamboo from construction, wood work industries the leaf, stem and a root part are able to use in production of activated carbon. In this work the bamboo stem were selected for production of activated carbon.

The bamboo (***Bambusa membranacea*)** used in this work was obtained from Southwest Ethiopia (tepi, district of yeki), were located in Lat-Long $7^{\circ}3'$N,$35^{\circ}18'$E with altitude of 1700m and the total annual rainfall 1678mm in a temperature maximum of 30°C and minimum of 15°C.

A large bamboo with small leaves, and a very branchy and bushy growth habit. The small leaves give it a 'fluffy' appearance which makes it look great when viewed from a distance. Culms are straight with weeping tops, 20–25 m high, 6–10 cm diameter, Covered with white powdery material.

Preparations of locally available matured bamboo were cut in to sticks, and mixed with proper size for production of powdered activated carbon suitable for the operations.

Fig. 3.1 Bamboo stems and Pieces of bamboo

Fig. 3.2 Chopped bamboos and the washing one

The chopped bamboo were washed by the running tap water until it removes some foreign matters on the surface of the bamboo and furher cleaning using a distelled water before going to oven dehydrating the moisture for 24 hr at 105°C. Mass of washed bamboo after compliation of dehydrationg process eache 100gm of sample ready for impergination with the activating agent pottassium hydroxide pellete, one to two ratio (pottassium hydroxide pellete:dry chopped bamboo) the chopped bamboo were soaked with the activator 4 hr and dry oven with 12 hr at 150° C (Evbuomwan, 2013). Most salt activating agents like zinc chloride has negative impacts its contributes the heavy metals zinc to the surrounding and acidic activating agent likes phospric acid its also has a draw back which is abale to relase phospates which is a nutrient its contributes for the formation of eutrification.

3.2.2 Carbonazation of the adsorbent

The impergenated dried samples (Chopped bamboo) were carbonaized at a carbonazation temprature of 500°C, 600°C and 700°C in *MF 106* Muffle Furnace with 3 hr, then carbon was cooled for 24 hr in a dissicators.

It was repeatedly washed with 0.1M Hydrochloric acid and distilled water until the washing was free from base medium it should be down to neutral media (pH *H183141 Romania* is 6-7).

Fig. 3.3 Washed and Grinded activated bamboo

Activated carbon washed by distilled water was again dried under thermally in a hot air oven at 105°C upon 12 hr (Gimba *et al*. 2004), thermally dried samples were crushed using a mortar and pestle to, obtained a desired a particles size (Sieves size 125 µm *model IC-205/EV).*

Fig. 3.4 Bamboos based activated carbon at 500°C, 600°C and 700°C

3.2.3 Characterizations of Adsorbent

The physical analysis of bamboo was conducted according to an American Standard Testing Method (ASTM). Proximate analysis of the adsorbent constituted determination of moisture content, volatile matter, fixed carbon, ash content and Fourier transform infra-red (FTIR) spectral analysis, scanning electron microscope and *XRD* were used to study the structural and morphological characteristics of adsorbents.

3.2.3.1 Moisture content

This method was used to determine the percentage of water in a sample by drying the BAC to a constant weight. The moisture content of the bamboo activated carbon was calculated by using digital moisture analyzer at 105°c for 24 hr Weighting 1g each, were used to determine the moisture content with the use of the digital moisture analyzer, the mean value of the three results is reported.

$$w = \frac{w_0 - w_d}{w_0} \times 100\% \quad \text{..... (3.1)}$$

Where w_o = original weight (g), w_d = weight after drying (g), w =moisture content (%)

3.2.3.2 Volatile matter determination

Empty crucible was weighed at the first; 1g of BAC was added in the crucible and weighed then after it was kept in the muffle furnace at a temperature of 900°C for 10min. Then taken out and kept in the desiccators for half an hour to cool down. The weight of the sample in crucible was measured again.

$$\text{Volatile content (\%)} = \frac{W_o - W_{ash}}{W_o} * 100\% \quad \text{..... (3.2)}$$

Where W_o is the dry weight of carbon sample before ash.

3.2.3.3 Ash content determination

A crucible was washed and dried in a hot air oven for 30min at a temperature of 105^0C. After heating, it was then cooled in a desiccator for 30min. The crucible was weighed using analytical balance. So that 1g of sample was weighed into the crucible and its weight was recorded. It was transferred to a muffle furnace maintaining a temperature of 650^0C for one hour, for complete de-carbonization to obtain white ash. The crucible and ash were cooled and the weight was recorded.

$$\text{Ash cont.} = \frac{W_{ash}}{W_o} \times 100\% \quad \text{..... (3.3)}$$

Where Wo is the dry weight of carbon sample before ashing, $W_{ash} = aftre\ ashing$

3.2.3.4 Carbon Yield

The total yields were determined after sample processing in terms of raw bamboo mass

$$Y_{ch} = \frac{w_{ch}}{w_0} \times 100 \quad \text{…… (3.4)}$$

Where $W_{o=}$ mass of bamboo carbon retrieved from the furnace, W_{ch}= mass of air-dried bamboo based activated carbon. (Pei-Hsing, 2014).

3.2.3.5 Bulk Density determination

The mass of Pycnomotor was measured at the first, fill the sample up to 50% volume of the pycnomotor and take the weight then fill the remain volume with distilled water, then calculate the bulk density.

3.2.3.6 Specific gravity

Specific gravity of bamboo based activated carbon can be calculated according to Udeh, N.U and Agunwmb, J.C. method.

$$G_S = \frac{M_2 - M_1}{(M_2 + M_4) - (M_1 + M_3)} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (3.5)$$

Where M_1 = Mass of empty pycnometer, M_2 = mass of oven dried AC inside the pycnometer, M_3 = mass of oven dried AC inside the pycnometer filled with water and M_4 = mass of pycnometer filled with water.

Most powder activated carbon specific gravity has been calculated by the above calculation in order to estimate the percent opening of the porosity of the adsorbent surfaces.

3.2.3.7 Porosity Evaluation

$$n = 1 - \frac{\rho}{\rho_w G_{s(1+w)}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (3.6)$$

Where n= porosity, ρ = bulk density of AC particles, ρ_w= density of water, $G_{s=}$ specific gravity of AC particles and W= moisture content.

3.2.3.8 Fourier Transformation Infrared Spectroscopy (FTIR)

The FTIR studies were performed to determine the various functional groups on the surface of the material (bamboo activated carbon before and after adsorption). The instrument used to record the FTIR spectra by FTIR instrument with kBr as reference. The spectrum was recorded by using FTIR spectrophotometric in a spectral range of 400-4000cm^{-1}.

3.2.3.9 Estimation of surface areas by iodine number

Iodine number is the mass (mg) of iodine (I_2) adsorbed from a standard 0.1N (0.05M) iodine solutions, when the equilibrium iodine concentration is exactly 0.02N (0.01M). According to the procedure defined by ASTM D4607-94(2006), for determination of iodine number 1g of activated carbon was added with 10ml of 5% HCl and swirled in a 250ml of conical flask until the entire activated carbon was wetted. The wetted solution was then boiled for exactly 30 seconds and the solution was cooled to room temperature. Then 100ml of 0.1N iodine solution was added to the content of the conical flask. This solution was filtered using whatman filter paper. Later 50ml of this filtrate were titrated against 0.10N sodium thiosulphate ($Na_2S_2O_3$) until the yellow color had almost disappears. 1ml starch indicator was added and titration was continued until the blue color just disappears. The equilibrium concentration was determined by calculation using the amount of sodium thiosulphate used in the titration.

$$I_n = X/M^* A \quad \text{(3.7)}$$

$$X = (12693N_1) - (279.246N_2V) \quad \text{(3.8)}$$

Where N_1=normality of iodine solution

$N_{2=}$normality of sodium thiosulphate solution

V=volume of sodium thiosulphate solution in ml

M=mass of activated carbon in g

A= correction factor depending on the residual normality N_r of the filtrate

$$N_{r=}\ N_{2*}V/50 \quad \text{(3.9)}$$

3.3 Solution preparation for the experiment

The wastewater (contaminant) that has contains (Pb^{2+}) ions artificially simulated in a laboratory. This was due to the fact that the effect of studying (Pb^{2+}) ions metal alone was required.

A lead stock solution with 1000 mg/L prepared from 1000ppm of was prepared by dissolving 1.5985g of lead nitrate, $Pb((NO_3)_2$ in distilled water and the volume was made to the mark in l000ml volumetric flask using distilled water and was used to prepare the working solutions with various initial lead concentrations by serial dilution.

In order to prepare a standard solution of 1g of lead it calculated from the total molecular weight of lead nitrate 1gm Pb = 207.2 and the total amount of lead nitrate is 331.2gm/mol therefore dividing the total value to the single element (Pb) its gives 331.2/207.2 =1.5985gm of lead nitrate were in 1000ml volumetric flask.

3.4 Batch Adsorption Experiments

Adsorption experiments were carryout under at room temperature under batch mode. Stock solution of lead (II) nitrate and metallic salts containing it was prepare suitably diluted with distilled water to investigate parametric effect of pH, dose of adsorbent and initial concentration.

$$\% \text{ removal} = \frac{C_0 - C_t}{C_0} * 100\% \quad \text{..... (3.10)}$$

Where: Co = initial conc. of adsorbate and Ct = finial concentration of the adsorbate after time (t)

3.5 Isotherm Study

Adsorption isotherms are used to describe the relationship between the amount adsorbed and its concentration in the equilibrium solution at constant room temperature.

The adsorption of Pb^{2+} were model using Langmuir, Freundlich,

$$\frac{C_e}{q_e} = \frac{1}{q_0 K} + \frac{C_e}{q_0} \quad \text{..... (3.11)}$$

Where: Ce is equilibrium concentration of the adsorbate (mg/l), q_e is mass of solute adsorbed per unit mass of adsorbent, q_o is a constant related to the adsorption capacity (mg/g) and K is experimental constant.

Linear form of Freundlich equation is given

$$\log(q_e) = \log K + \frac{1}{n}\log C_e \quad \text{..... (3.12)}$$

Where: C_e is equilibrium concentration of the adsorbate, q_e is mass of solute adsorbed per unit mass of adsorbent, k and n are Freundlich constants representing the adsorption capacity of adsorbents and the intensity of the adsorption, respectively.

3.6 Adsorption experiment to determine the optimum experimental Parameters

Table 3.1 Experimental Parameters

Parameters	**Condition needs to investigate**
pH	3,5 and 6
Adsorbent dose (gm)	1,1.5 and 2
Initial metal ion concentration (mg/l)	50, 100 and 150

The parameter were selected at higher initial concentration of metal ion this is because of the real wastewater that has been taken from the paint industry has contain the lead metal ion above the standard level (30ppm) and it doesn't have a proper treatments plants, therefore the situation derives us to investigate the contaminant under high ion concentration to see the adsorption performance of the bamboo activated carbon. The dose at the same time it provides the large surface areas which is the potential of provided additional functional groups responsible for the adsorption of lead metal ions.

The pH mainly revolves around acidic media mostly the contaminant lead ion adsorption more successful in acidic condition. An increase in pH beyond 6, the amount of hydroxyl ions increased in the solution and metal ions react and precipitate with OH^- ions which results impossible the biosorption process (Chimie, 2014). That's why the condition mainly sticks with acidic media and seeing the adsorption performance of locally produced bamboo activated carbon.

4. Results and Discussions

4.1 Physicochemical analysis of bamboo based activated carbon

The physicochemical characteristics of bamboo activated carbon were investigated by American Standard test method (ASTM) and the data were tabulated shown below in table 4.1

Table 4.1 physicochemical property of bamboo activated carbon

Properties	**Carbonization temperature(°C)**		
	500	600	700
Iodine number (mg/g)	814.69	802.45	789.01
Porosity	0.704	0.6962	0.6926
Moisture content (%)	7.7	7.01	6.20
Ash content (%)	3.51	4.73	5.53
Yield (%)	40.6	38.04	36.01
Volatile matter (%)	23.4	22.27	21.07
Specific gravity (gm/cm^3)	1.470	1.486	1.550

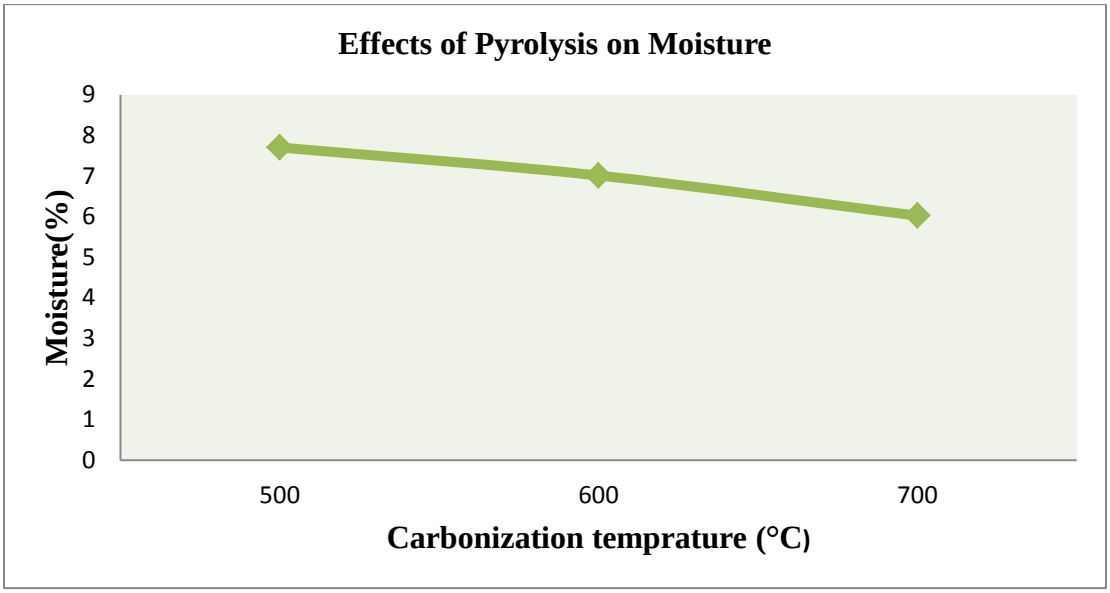


Fig. 4.1 Effects of carbonization temperature on moisture.

The moisture content (7.70% to 6.20%) of bamboo activated carbon directly influences the carbonization time and carbonization temperature. The drying period of bamboo carbonization will prolong if the moisture content is too high and as a result, the carbonization process will extend with more electricity consumption. The lower moisture content of bamboo speeds up the bamboo carbonization process.

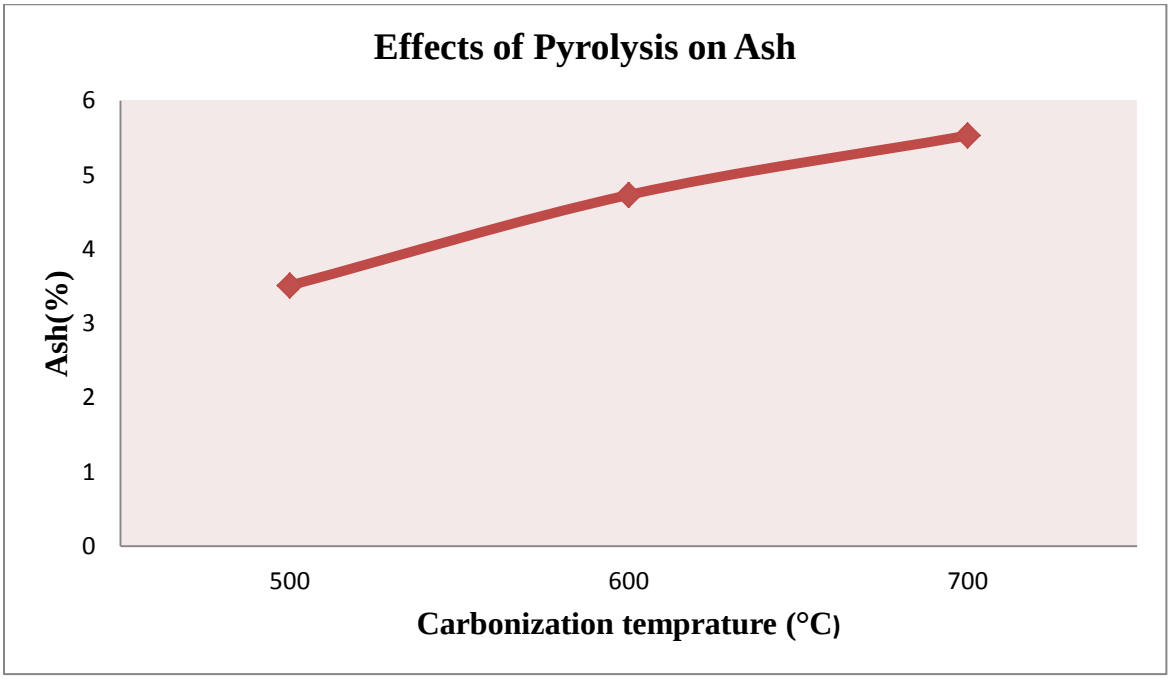


Fig. 4.2 Effects of carbonization temperature on Ash

The ash of bamboo activated carbon is its inorganic constituent, which is white substance after bamboo carbon has burned completely at high temperature (650^0C), the ash percentage in bamboo carbon increase from 3.51 to 5.53 % with the rising of carbonization temperature. The ash of bamboo carbon is positively proportional to the temperature of carbonization. The low ash content a positive factor the AC production, since the mineral matter, because of hydrophilic characteristics, promote the water adsorption (Paulo *et al*. 2017). The result showed a good agreement with previously worked paper 5.55% (Masood *et al*. 2015).

The yield rate is inverse to the temperature of carbonization, that is, the higher carbonization temperature, the lower yield rate.

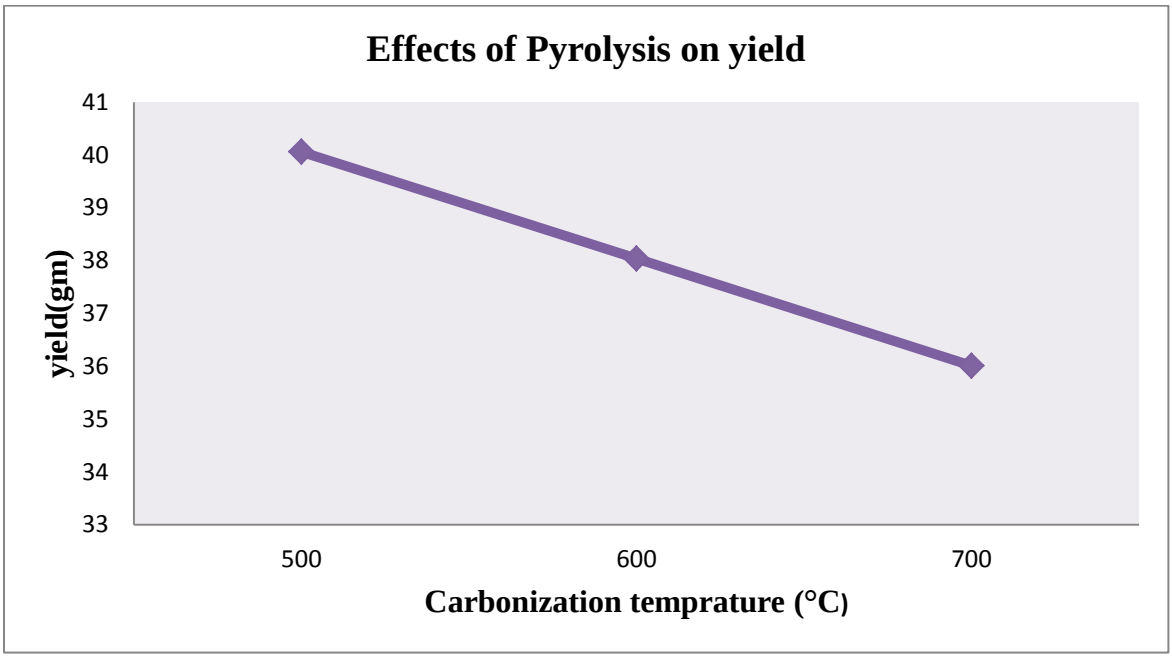


Fig. 4.3 Effects of carbonization temperature on yield

The maximum porosity development while still minimizing the material's loss due to an excessive combustion, the yield percentage decrease with an increasing activation temperature, this means increasing reactivity, ascribes a significance amount of volatile matter released during carbonization, but at the same time increasing the volume of pore present with a certain limit extent of carbonization temperature.

4.1.1 Porosity

Porosity is a measure of the void fraction in a material. Voids either be 'closed', and inaccessible or 'open' and connected to other voids and hence to exterior of the material. Porosity can be modified or changed by a variety of process during the test such as deformation hydrothermal alteration (Lawrence, 2017). The porosity were calculated by different carbonization temperature the result has been shown that the decreasing tendency of percent porosity the fact that the carbonization temperature increases inversely correlated with that of percent porosity of bamboo activated carbon.

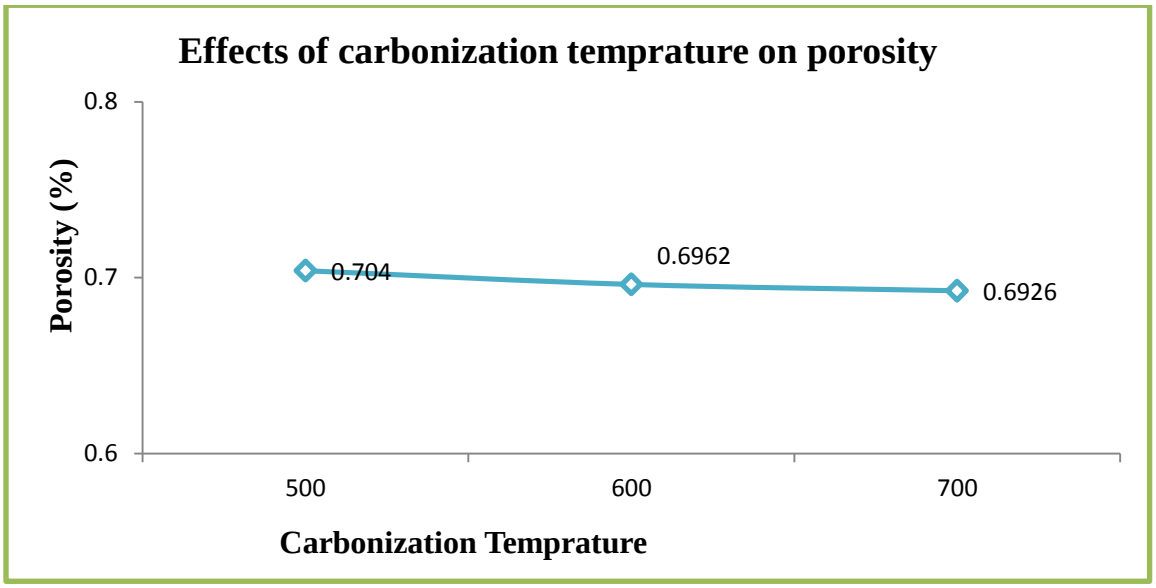


Fig. 4.4 Effects of carbonization temperature on porosity

4.1.2 Scanning Electron Microscope (SEM)

Scanning electron microscope (*model* INSPECT F 50 at 20Kv energy supply) was used to know the surface texture and porosity of the sample (bamboo activated carbon) in secondary electron imaging mode the unloaded shows that the presence of an organized pore region in the fibrovascular bundle wall after the activation that permits the diffusion of the contaminants in a faster way to the inner regions of the produced bamboo/KOH. (Before adsorption fig 4.5) amorphous structures without definite shapes and a loaded (fig 4.6 with contaminant) sample has been investigated.

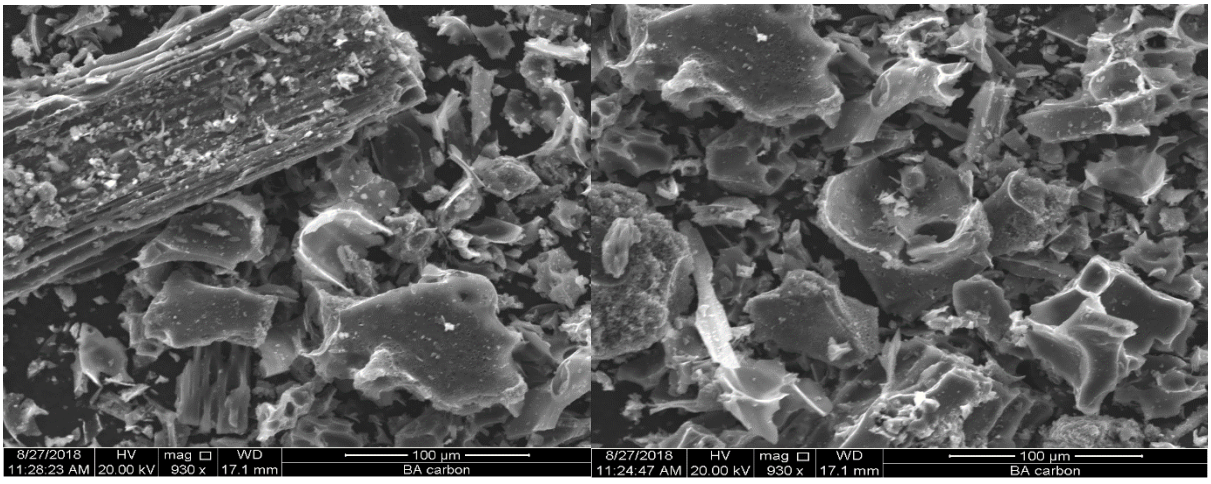


Fig. 4.5 SEM micrograph of BAC at 950x magnification (before contaminant loaded)

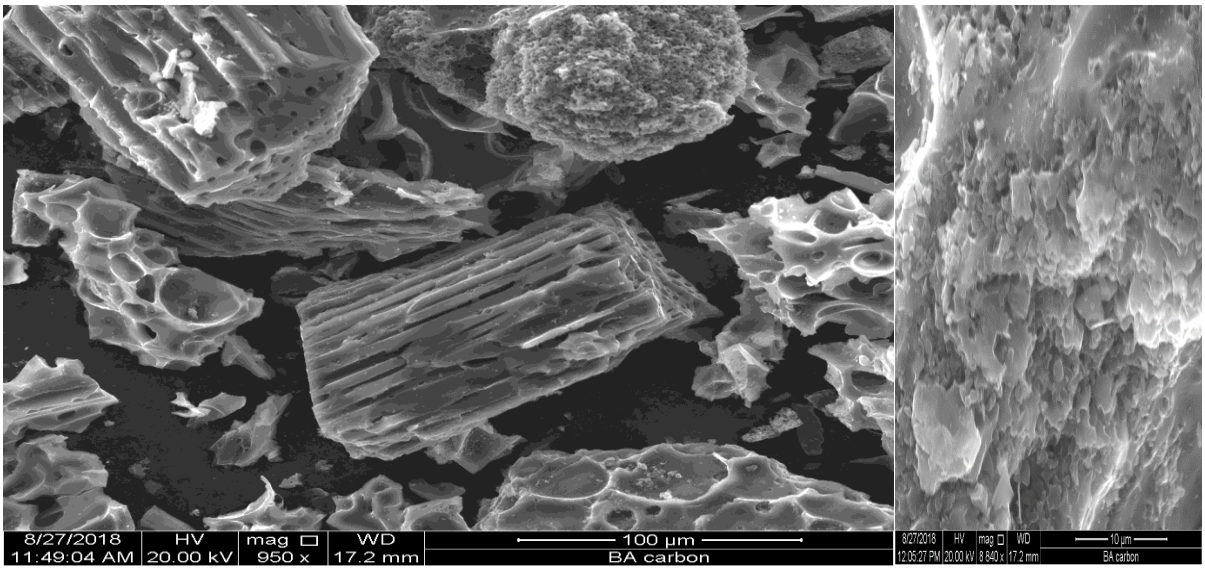


Fig. 4.6 SEM micrograph of BAC at 950x magnification (After adsorption)

Fig 4.6 shows changes at some parts of the surface particles with native biosorbent (bamboo activated carbon) which depicts that Pb^{2+} ion have deposited as aggregates in the biosorbent and still have a very porous nature of some parts of the absorbent surface, still support the accumulation of contaminant on the pore site.

4.1.3 Fourier Transformation Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (*model* IRAffinity-1S) were used to identify the functional group of bamboo activated carbon 2mg of bamboo activated carbon powdered mix with 300mg of anhydrous KBr (potassium bromide) the mixture was pressed under vacuum (hydraulic press Model-15) the spectra were performed between 4000 and 400cm^{-1} for the evaluation of surface composition of the bamboo activated carbon.

The band at 3400 cm^{-1} corresponds to the C-H vibrations in Alifatic, olefinic, and aromatic hydrocarbons groups and 1770-1650cm^{-1} corresponds to vibration in C=O Carbonyl causes the emergence of the bands while 1700-1600 cm^{-1} corresponds to C=C Olefinic structures bands, 1480-1420 $cm^{-1}$to C-H Alifatic structure bands, 1430-1360cm^{-1}Bending O-H and C-H Hydroxyl, carboxylic acid Olefins, methyl, 1120-1070$cm^{-1}$to Stretching C-O Secondary Hydroxyl and 1060-1000 cm^{-1} Stretching C-O Primary Hydroxyl.

With increasing pyrolysis temperature, the bands assigned to O–H stretching vibration (near 3402.7 cm^{-1}).The intensity of the band at 1690–1591.73 cm^{-1} (aromatic C=C stretching and C=O Stretching of conjugated ketones and Quinone's) also diminished with increasing temperature 873 cm^{-1} (out–of–plane deformation by aromatic C–H groups) might be caused by carbonates. The sharp peaks at 1248.6–1000 cm^{-1} were assigned to C–O and C-C stretching, and these peaks markedly decreased probably due to the loss of polysaccharides during pyrolysis.

Table 4.2 Fourier Transformation Infrared Spectroscopy wave number

Wave number (cm^{-1})	**Assignment**
3402.7	O-H stretching in hydrogen bond.
3300-3200	=C-H stretch
1624	C=C Alkane, C=O
1170.84, 1072.47	C=O stretch, C-O Alcoholic stretch, carboxylic acid, anhydrides
873.79,750.	Aromatic (out-of plane band)

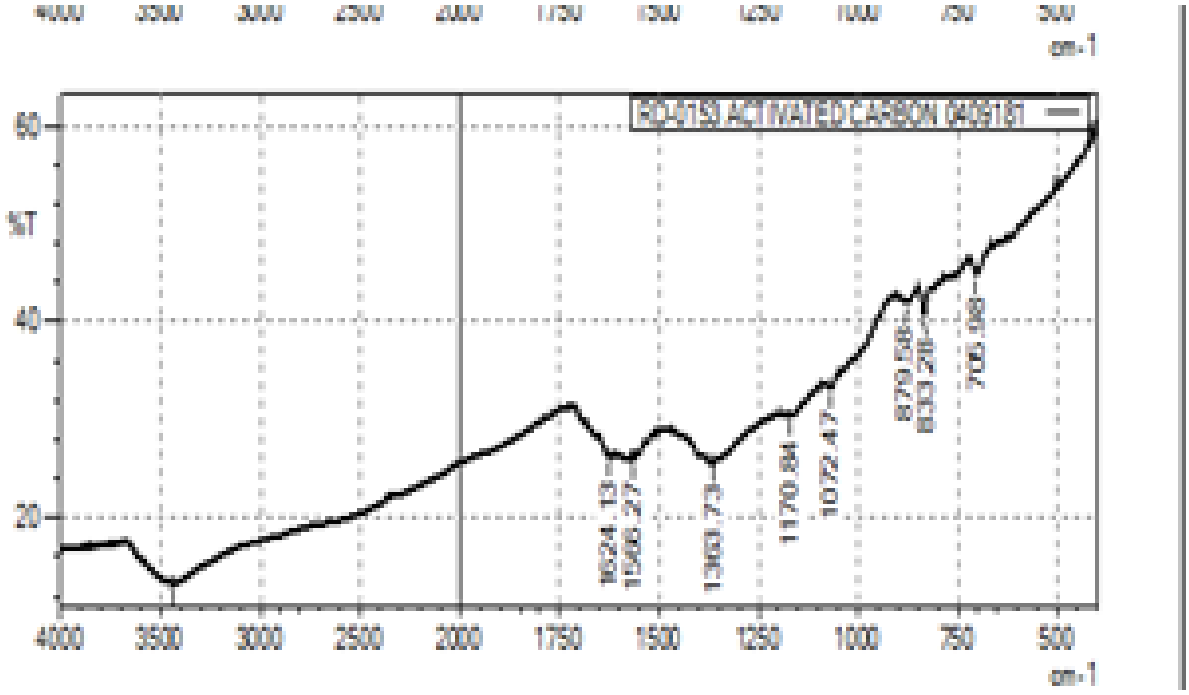


Fig 4.7a FTIR spectra for BAC before adsorption.

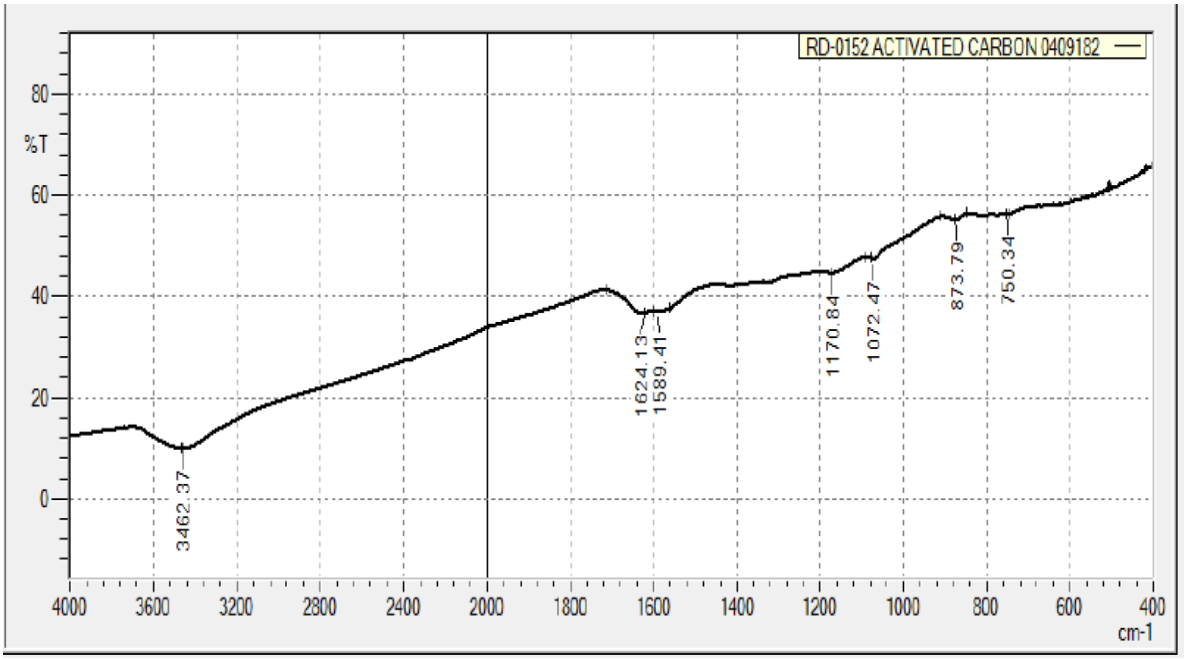


Fig. 4.7b FTIR spectra for BAC after adsorption.

It can be seen that the IR-spectra indicated the presence of ionisable functional groups (negative ions contains functional groups); their ionization leaves vacant sites which can be replaced by metal ions. This gives an indication that those materials could be used as adsorbents for heavy metal (Pb^{2+}) removal.

4.1.4 XRD (X-ray diffraction)

X-ray diffraction was used to evaluate the existence of amorphous and crystalline between carbon matrixes. The XRD spectra of bamboo activated carbon are shown fig 4.8 According to the powder diffraction file based on *XRD* data base obtained from (*miniflex 600 PxRD)* were evaluated by *origin8 software*. The diffraction spectrum of bamboo activated carbon has amorphous in nature and did show some obvious crystalline peak at the scan range *(0 to 2Θ);*

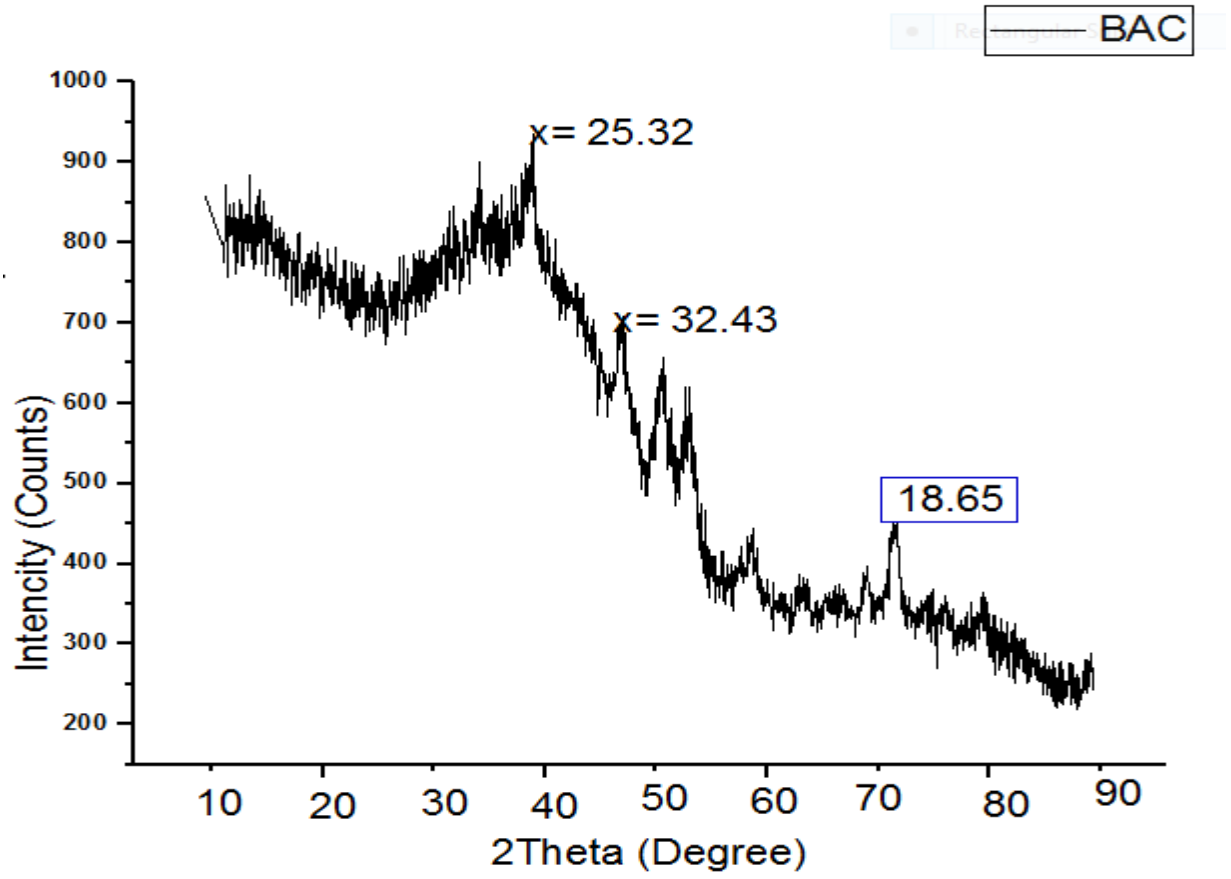


Fig. 4.8 XRD result of bamboo activated carbon

The XRD pattern shows three broad intense peaks at 35^0(x=25.32), 45^0 (x=32.43), 72^0 (x=18.65) respectively shows, were the bamboo activated carbon has a formation of nanocrystalline structures on the matrix, *corr_pos_value_RS1 "1.250deg" *corr_pos_value_RS2 "0.3mm" however if the main information desired is weather peak originates from amorphous carbon or semi graphic carbon, an approximation of its peak width at half peak intensity should be made, if this width is greater than 0.5deg" using copper radiation (that is the carbon has an average crystalline size of less than 150Å) the carbon considered as amorphous. If the width is less than 0.5deg" an estimation of the angle at maximum peak intensity must be made to determine the graphite carbon content (Walker *et al*. 1955).

4.1.5 Iodine Number

The effect of iodine number adsorption on bamboo activated carbon result showed, the reduction in adsorptive capacity of the carbons values is an indication of the beginning of structural deformation of the already produced bamboo activated carbons.

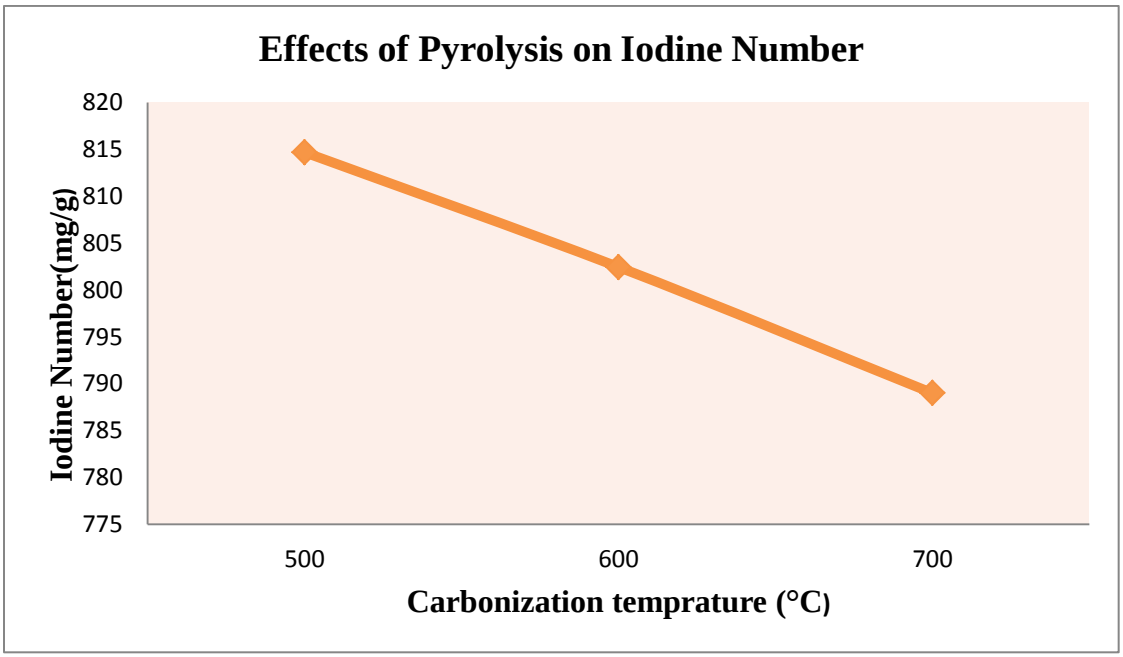


Fig. 4.9 Effects of carbonization temperature on iodine number

It is evident that the activation temperature had a significant influence on the iodine number. The iodine number was observed to decrease at higher activation temperatures. Around 600°C and above the iodine number practically decreases. The iodine values were significantly affected by activation temperature and activation time.

According to (Prakash *et al*. 2006). Increase in activation time up to 2 hr. showed that the iodine number increase slowly when the activation temperature 500°C and 600°C iodine was observed with a maximum value 765mg/g. further increase activation time and temperatures leads to complete burn off due very high gasification rate. In this studies the iodine number figures were shows the decreasing tendency this may be a result of activation times it holds 3 hr this may accelerate the gasification process and a certain degree breakage of internal structures.

4.2 Effect of pH of Adsorbate Solution

P^H effect on sorption of lead was conducted between the P^H of solution ranges of 3-6. An increase in pH beyond 6, the amount of hydroxyl ions increased in the solution and metal ions react and precipitate with OH^- ions which results impossible the biosorption process (Chimie, 2014). Lowering the pH below 2 of the solution probably results in decreasing of biosorption levels and this can be explained due to competition between protons and metal ion for capturing same available sites of the adsorbent, which at low pH, metals (Pb^{2+}) ions do not successful.

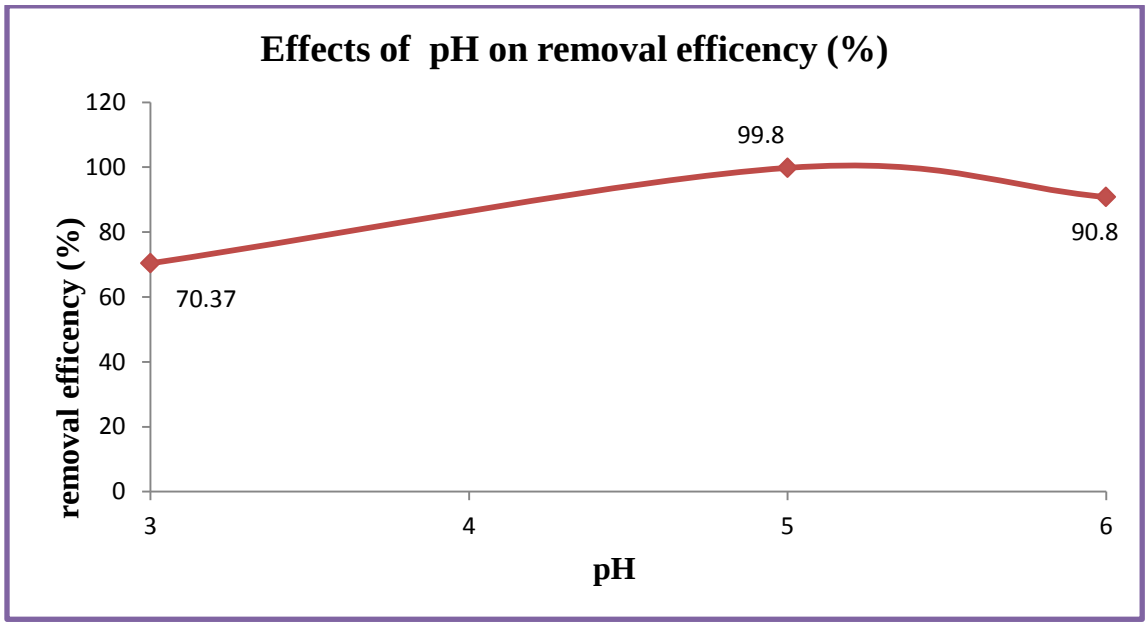


Fig. 4.10 Effects of pH on the removal efficiency

From the above fig 10 the efficiency smoothly started to increase in pH 3 the increase in metal removal was observed as pH increases. This may be due to decrease in competition between hydronium ions and metal ions for the surface sites when the pH at 5 it reaches the maximum point 99.8 % removal efficiency This is also by the decrease in positive surface charge on the adsorbent, which resulted in a lower electrostatic repulsion between the surface and the metal ions and hence uptake of metal ions get increased, however the pH closed to 6 decline at alarming rate this condition illustrate the surface of bio absorbent (bamboo activated carbon) were crate a competent site between the positive metal (Pb^{2+}) ions and hydroxyl ion which means OH^-ions increment causes a decrease in adsorption of metal ions at adsorbent adsorbate

interface, for metal adsorption on different adsorbent. It is also supported in an alkaline medium lead ions tend to hydrolyze and precipitate instead of adsorption on adsorbent. It was deteriorated with accumulation of metal ions, and making impossible true adsorption.

4.3 Effects of initial metal ions concentration

The initial metal ion concentration provides an important driving force to overcome all mass transfer resistances of metal ion between aqueous and solid phases.

Increase in metal ions concentration results in increase in number of available molecules per binding sites of the adsorbent. Increase in initial metal ions concentration enhances the interaction between the metal molecules and the surface of the adsorbent (Itodo *et al*., 2014) meaning, it provides a driving force to overcome mass transfer resistance between the biosorbent and biosorption medium.

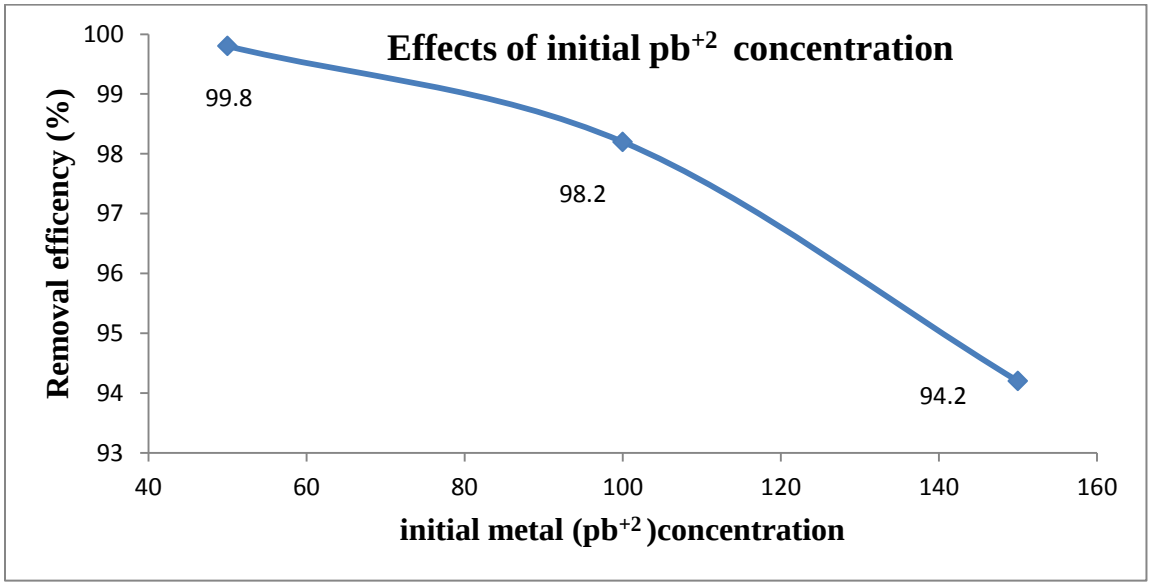


Fig. 4.11 Effects of initial concentration on removal efficiency

The capacity of the adsorbent increased significantly even though there is slight decrease in the adsorption efficiency with the increment of initial concentration. The increase of capacity can be due to increment of driving force that is concentration gradient, which causes an increase in the number of metal ions coming in contact with the adsorbent. On the other hand, the number of available adsorption sites in adsorbent is the same for all initial concentrations; thus the initial

concentration increases with more number of ions and the same change to be adsorbed and competes the same adsorption sites. This may cause to leave many ions without being adsorbed and to decrease the efficiency of the removal upon increases the concentration of (Pb^{2+}) ions.

4.4 Effects of Dose on Removal Efficiency

Adsorption of lead increased from 97.90 to 99.8% with increasing adsorbent dose from 1gm/0.250l to 2gm/0.250l, respectively. This is because for a constant initial lead concentration, while increasing the adsorbent dose provides a greater adsorption surface area, on the other hand metal uptake per adsorbent unit versus adsorbent dose shows that the capacity high at low dose and vice versa.

This outcome can be attributing to the fact that some of the adsorption sites remain unsaturated after the bamboo activated and the contaminants were together. There might be the formation of particles aggregation, resulting in a decrease in the total surface area and an increase in diffusion path length, which contribute to decrease in amount adsorbed per unit mass.

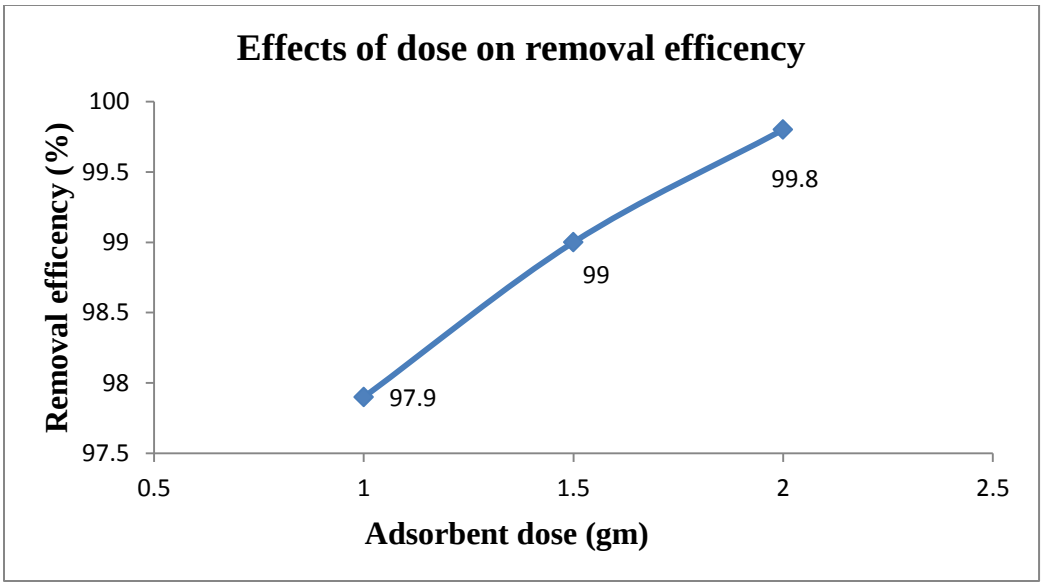


Fig.4.12 Effects of dose in metal uptake efficiency

4.5 Effect of Pb^{2+} concentration and pH (concentration and pH) versus % removal

In order to study the interaction among different independent variables and their corresponding effect on the response variable, contour plots were drawn. A contour plot is a graphical representation of a three dimensional response surface as a function of two independent variables, maintaining all other variables at fixed or different level. This can be helpful in variable. Interaction effect of initial Pb^{2+} ion concentration and pH was shown in contour and 3D plot.

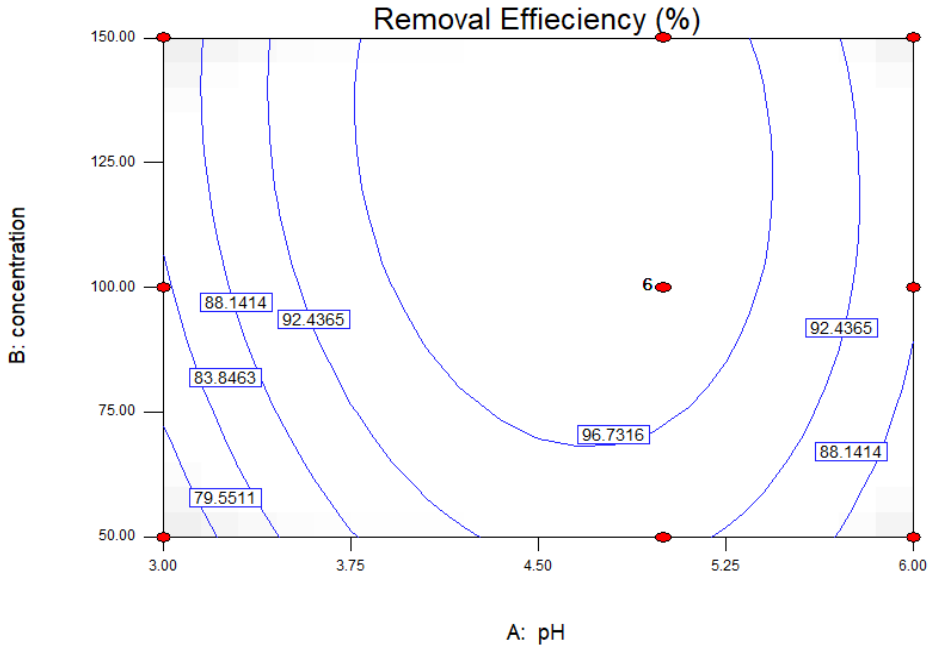


Fig 4.13 contour plots showing the combined effect of Pb^{2+} concentration and pH at 2g dose, shaking time 120min.

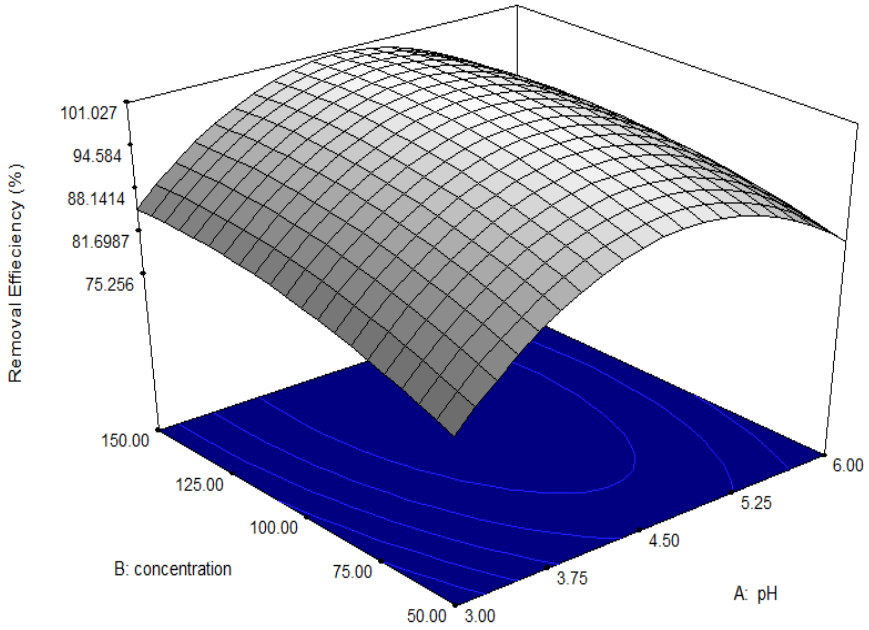


Fig 4.14 3D plots showing the combined effect of Pb^{2+} concentration and pH at 2g dose, shaking time 120min.

In general at higher Pb^{2+}concentration, the percent removal decreases at the pH decrease from 6 to 3 while at lower the concentration % removal increase.

4.6 Effects of independent variables (Dose and pH) versus % removal

The removal efficiency increase from 97.90 to 99.8% with increasing adsorbent dose from 1gm/0.250l to 2gm/0.250l, respectively. This can be attributed to increased number of sorption sites available for sorbent solute interaction, with further increase of adsorbent dose from 1gm to 2gm.

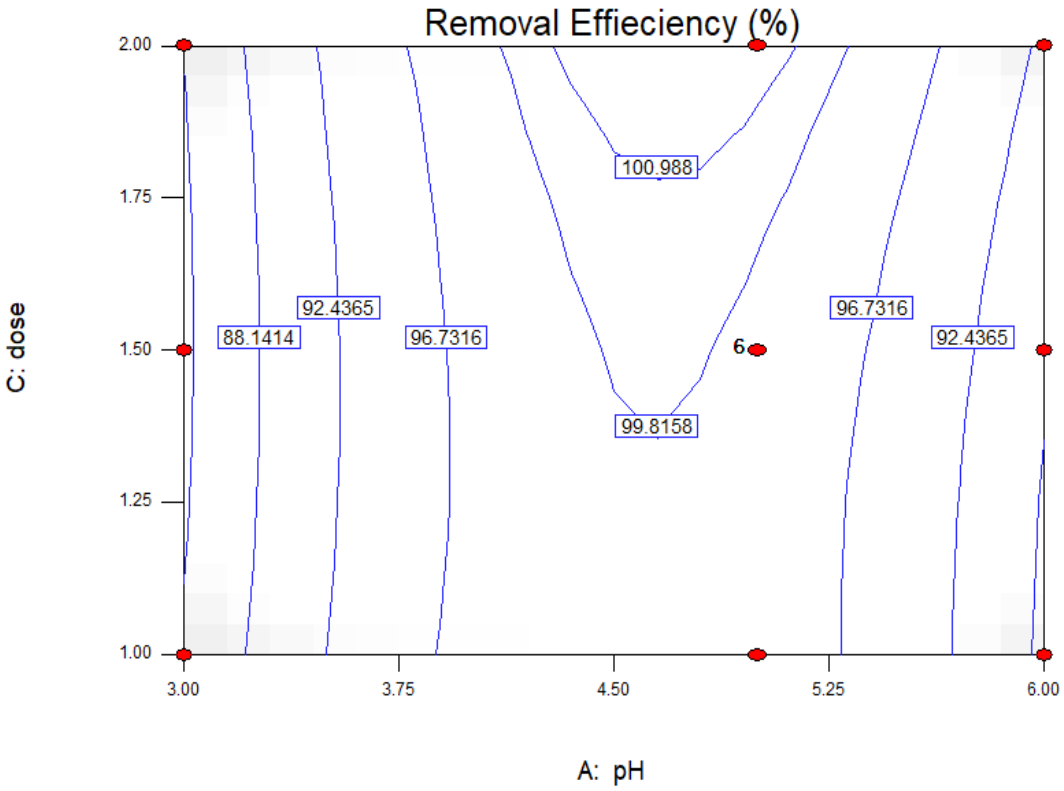


Fig 4.15 contour plots showing the combined effect of Pb^{2+} dose and pH at 2g dose, shaking time 120min.

According to, (Wasewar *et al*, 2009) increase in adsorption with adsorbent dose can be attributed to increased adsorbent surface area and availability of more adsorption sites.

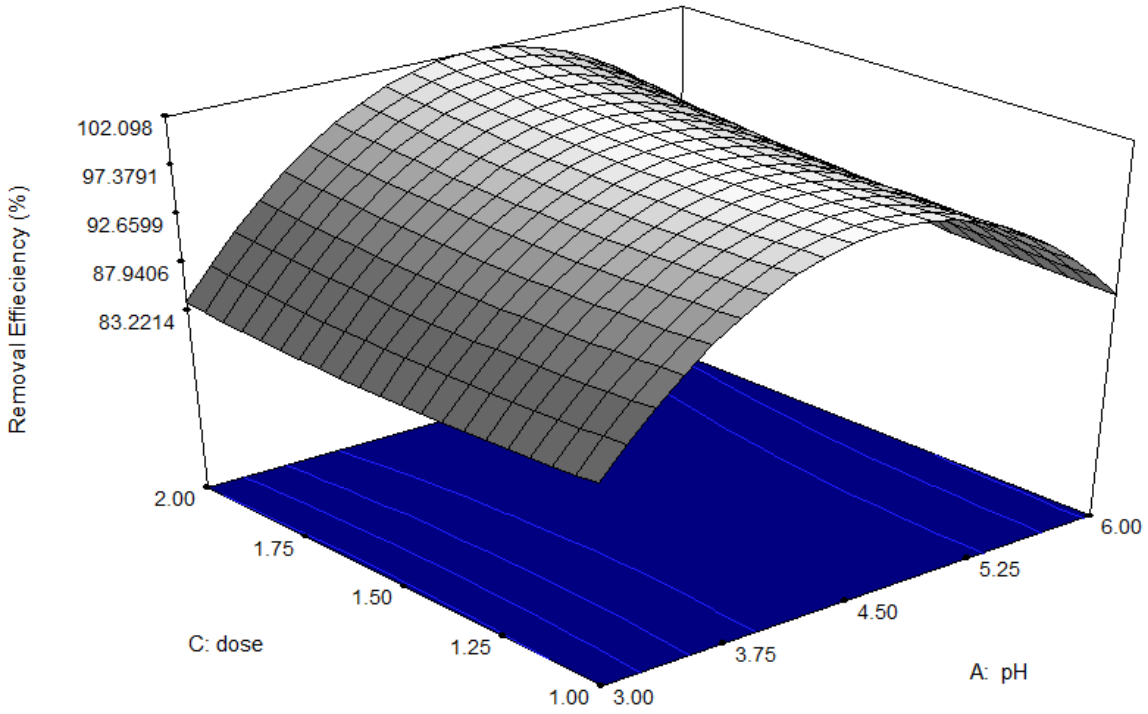


Fig 4.16 3D plots showing the combined effect of Pb^{2+} dose and pH at 2g dose, shaking time 120min.

4.7 Analysis of variance

Table 4.3 ANOVA results of the regression model for optimization Pb^{2+} adsorption.

Source model	Sum of squares	DF	Mean square	F value	Prob < F	
	1975.06	9	129.45	14.79	<0.0001	Significance
A (pH)	134.75	1	134.75	9.08	0.0064	
B(mg/l)	218.30	1	218.30	14.72	0.0009	
C (dose)	18.52	1	18.52	1.23	0.2759	
A^2	1057.08	1	1057.08	71.26	<0.0001	
B^2	62.05	1	62.05	4.18	0.0530	
C^2	5.67	1	5.67	0.38	0.5429	
AB	28.41	1	28.41	1.92	0.1802	
AC	16.57	1	16.57	1.12	0.3020	
BC	2.52	1	2.52	0.17	0.6842	
Residual	326.36	22	14.83			
Cor total	2301.42	31				

The Model F- value of 14.79 implies the model significant. There is only a 0.01% chance that a "Model F- value" this large could occur due to noise. Values of "Prob < F" less than 0.0500 indicate model terms are significant. In this case A, B, AB, AC are significance model terms. Values greater than 0.1000 indicate the model terms are not significance. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

4.8 Equilibrium sorption study

Sorption studies describe the interaction of adsorbates with adsorbent, and established equilibrium between adsorbed metal ions and the residual metal ions in solution during the surface sorption. The interaction between adsorbate and adsorbent is characterized using adsorption isotherm models**.** Adsorption isotherms are mathematical models that describe the distribution of adsorbate species among liquid and adsorbent. Based on a set of assumptions, that is mainly related to the heterogeneity or homogeneity of adsorbents, type of coverage and possibility of interaction between adsorbate species**.**

4.8.1 Langmuir isotherm model

Langmuir‘s isotherm calculated using equation stated in methodology section and the result were tabulated in table 4.4 below.

Table 4.4 Langmuir isotherm model for adsorption of Pb^{2+} using BAC.

Optimum condition	C_e	Q_e	$1/Q_e$	C_e/Qe	$1/C_e$
pH 5, 2gm	1.0719	6.116	0.1635	0.1752	0.9329
	1.7581	12.28	0.0814	0.1431	0.56879
	2.1205	18.48	0.054	0.0648	0.4715

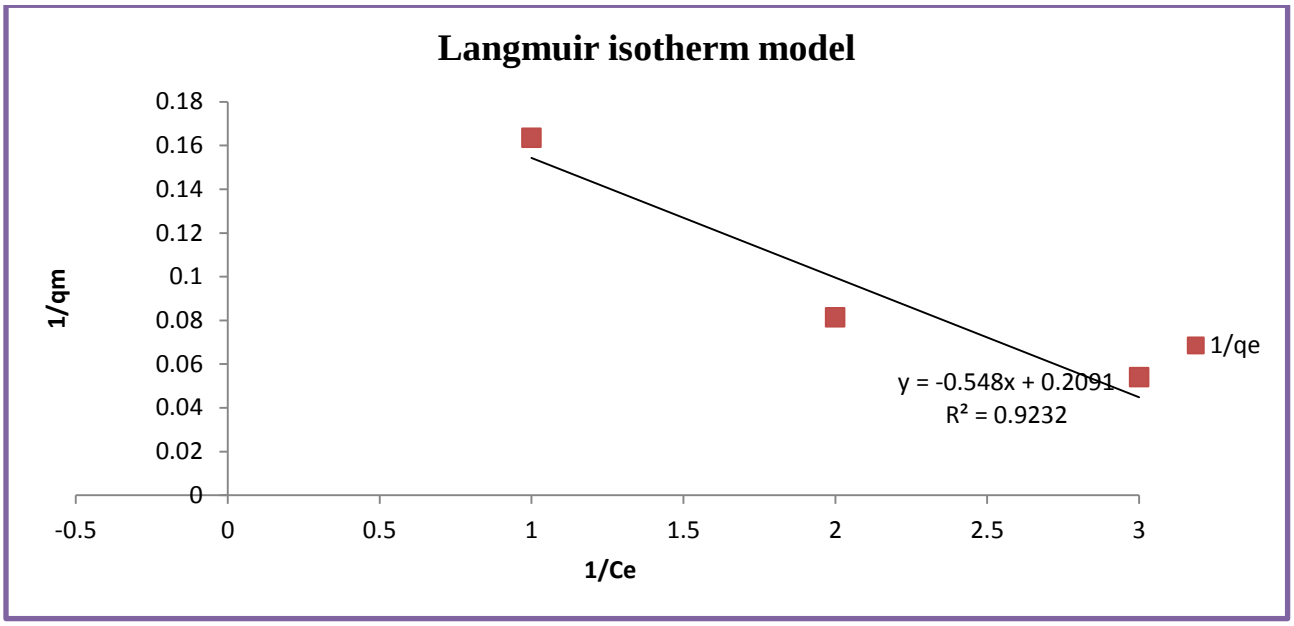


Fig. 4.17 Langmuir adsorption isotherm for adsorption of Pb^{2+} on BAC pH; 5, room temperature, adsorbent dose: 2gm and 120 minutes contact time**.**

The plot has value of 0.9232 with a slope and intercepts values of 0.0548 and 0.2091 respectively. The values of the Langmuir constants (b (0.49) and Q_o(4.04)) for the adsorption of Pb^{2+} ion using bamboo activated carbon were determined from the slope and intercept values.

4.8.2 Freundlich isotherm model

Freundlich isotherms are displayed in a logarithmic scale, by taking the logarithmic equation by plotting the $logQ_e$ versus $logC_e$ in order to determine the Freundlich isotherms constants.

Table 4.5 Freundlich isotherm model for adsorption of Pb^{2+} using BAC.

Optimum condition	C_e	Q_e	$1/Q_e$	C_e/Qe	$logQ_e$	$logC_e$
pH 5, 2gm	1.0719	6.116	0.1635	0.1752	0.7864	0.030
	1.7581	12.28	0.0814	0.1431	1.0891	0.2450
	2.1205	18.48	0.054	0.064	1.2661	0.3264

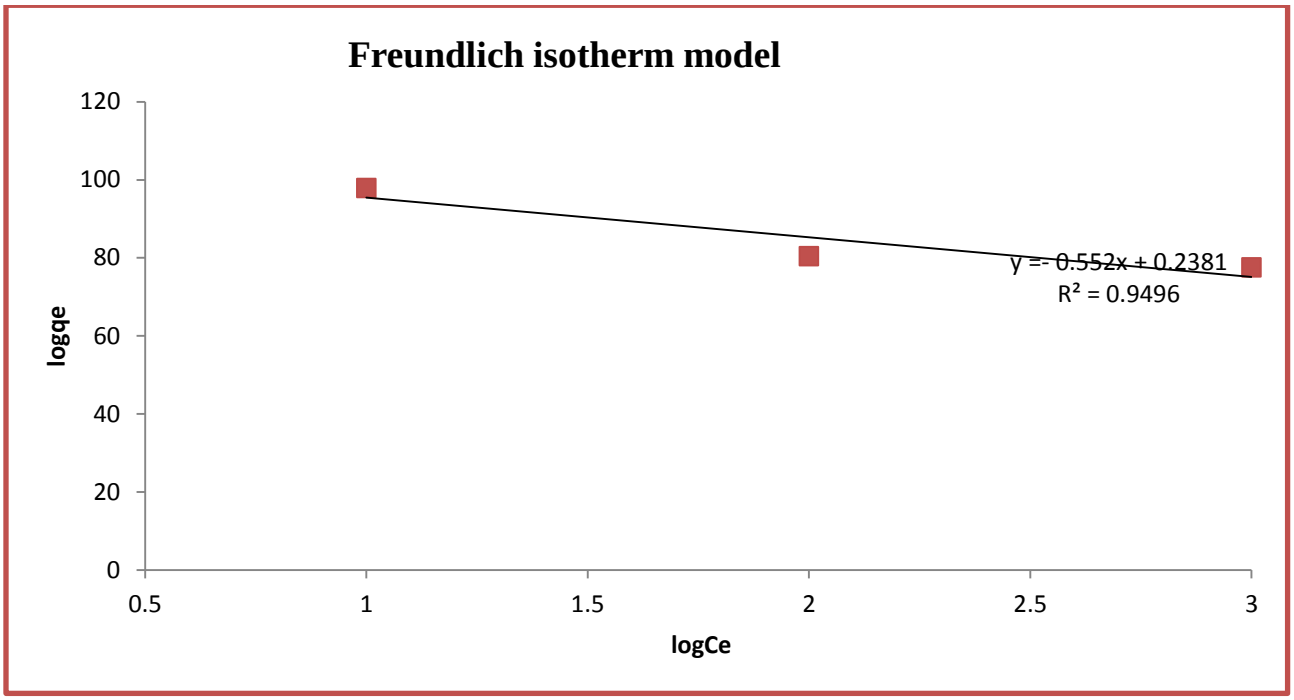


Fig. 4.18 Freundlich adsorption isotherm for adsorption of Pb^{2+} on BAC pH; 5, room temperature, adsorbent dose: 2gm and 120 minutes contact time**.**

The values of the equilibrium concentration C_e the amount adsorbed Q_e and the logarithm of C_e and Q_e at the various initial concentration of lead ion. The value were achieved by keeping other parameters constant varying the concentration 50mg/l to 150mg/l. fig 4.18 shows linear plot of log C_e and log Q_e with R^2 value of 0.9496, with a slope and intercept of 0.0552 and 0.2381 respectively.

The Freundlich parameters (K_f and n) for adsorbtion of lead ion into bamboo based activated carbon are determined from the slope and intercept values of the equilibrium data well fitted in this isotherm as compared to Langmuir isotherm which means it had a greater R^2 value, indicating that the sorption of Pb^{2+}metal ions into the studied adsorbents was favorable. Using the Linear Least-Squares Regression model the constants were evaluated (K_f (1.08) and n (1.824)).

From the high value of coefficient of regression R^2 =0.9496 obtained for the adsorption of metal ions Pb^{2+} it was clear that adsorption of lead onto bamboo activated carbons derived from these adsorbents obey Freundlich isotherm model. In addition most literature states that the decreasing

trend of a graph shows that the interaction between contaminant and BAC surface had a chemosorption natures.

4.9 Effects of Carbonization temperature on removal efficiency

The maximum porosity development occur during the optimal pyrolysis process, when the pyrolysis process carried out at the elevated temperature depends upon whether the agricultural products or not the naturally existed amorphous carbon structure has been distracted, the situation leads unfavorable adsorption condition or weak performance of the products.

Under this work besides investigating the physicochemical properties of the bamboo based activated carbon has been done at different carbonization temperature.

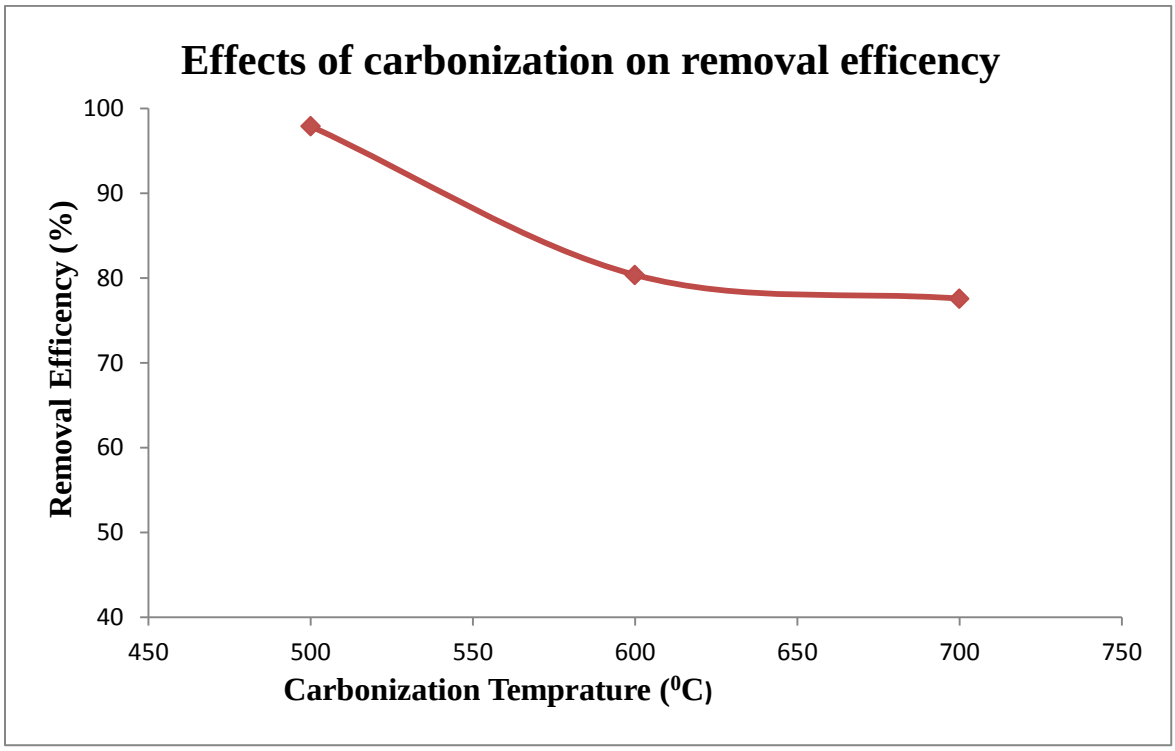


Fig. 4.19 Effects of carbonization temperature on removal efficiency

Initial metal ion concentration (100mg/l), average dose of 2gm at optimal pH at 5 were selected, According to the investigation of this different temperature zone of product revealed from the above fig 4.19 shows the removal efficiency were down from 97.9% to 77.5%, this may be the porosity of the bamboo activated carbon decreases from 0.7040% to 0.6926% which is due to the thermal influence of carbonization temperature and it may also the lack of active site on the

adsorbent. Availability surface area has a positive relation with adsorption process once the available surface area has influenced, the contaminants are unable to absorb into the existed surface area.

4.10 Removal efficiency of bamboo activated carbon for real waste water

Attempts were made to apply laboratory experimental results to real wastewater samples in order to move from the experiment to the application in real world. Wastewater sample lead was obtained from Nefas silk paint industry which is located in Addis Ababa.

Batch sorption test was conducted for lead metal at the optimum points obtained from prior synthetic wastewater batch sorption experiment. The feasibility and efficiency of a biosorption process depends not only on the properties of the biosorbents, but also on the composition of the wastewater. Taking this statement into consideration Pb^{2+} metal ion in wastewaters were taken from mixing point as they are discharging to investigate other metals ions interference for sorbent sites. Initially the effluent has contains 29.950ppm (approximately 30mg/l) of lead ion concentration so that the optimum condition used to see the effects on the real world applications.

Table 4.6 Optimum parameter for real wastewater (Nefas silk paint industry) application**.**

Metal ion	**Optimum parameter**			**Real wastewater parameter**	
	pH	Contact time (min)	BAC dose (gm)	pH	Initial con. (mg/l)
Pb^{2+}	5	120 min (2hr)	2	5 (after adjusting)	30ppm

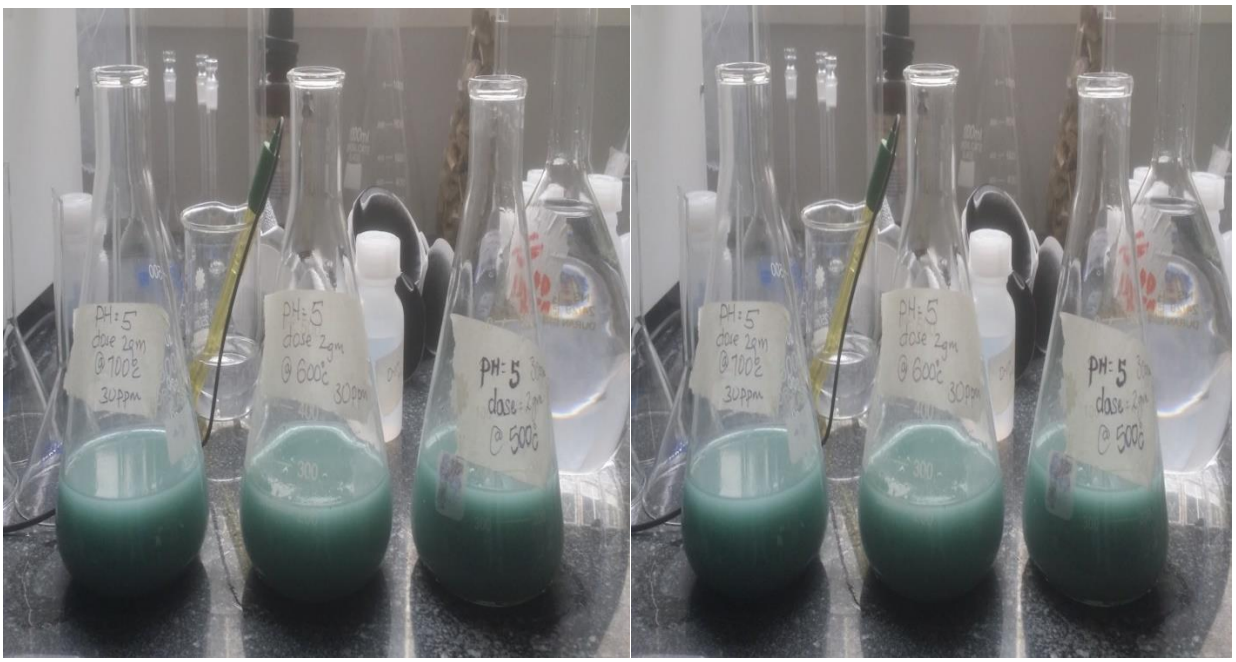


Fig. 4.20 Real wastewater from Nefas Silk paint industry

After adsorption of waste water containing Pb^{2+} metal ion onto bamboo activated carbon, it was found that lead reduction was observed at each carbonization temperature.

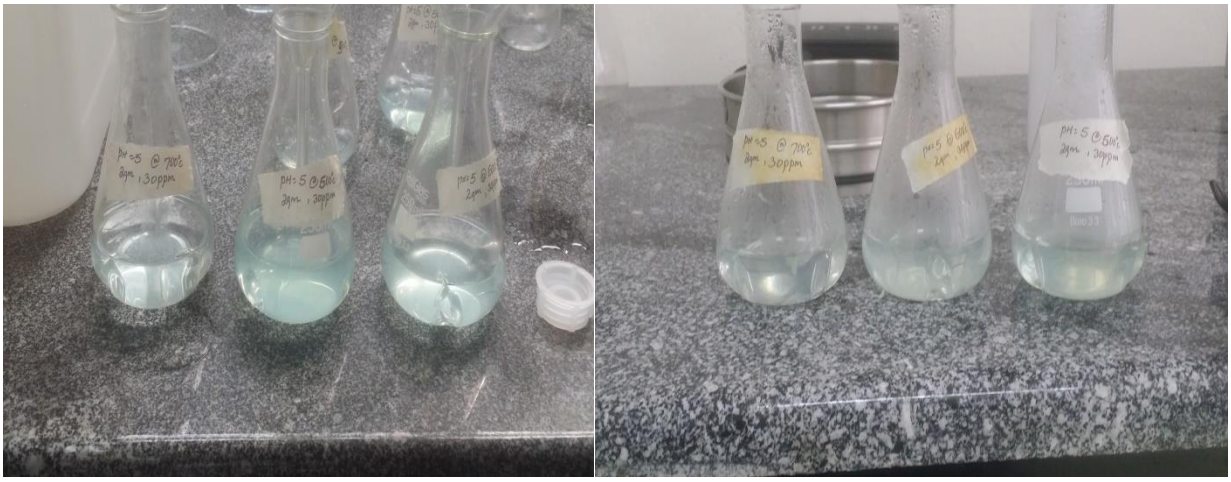


Fig. 4.21 After adsorption of the real wastewater by bamboo activated carbon**.**

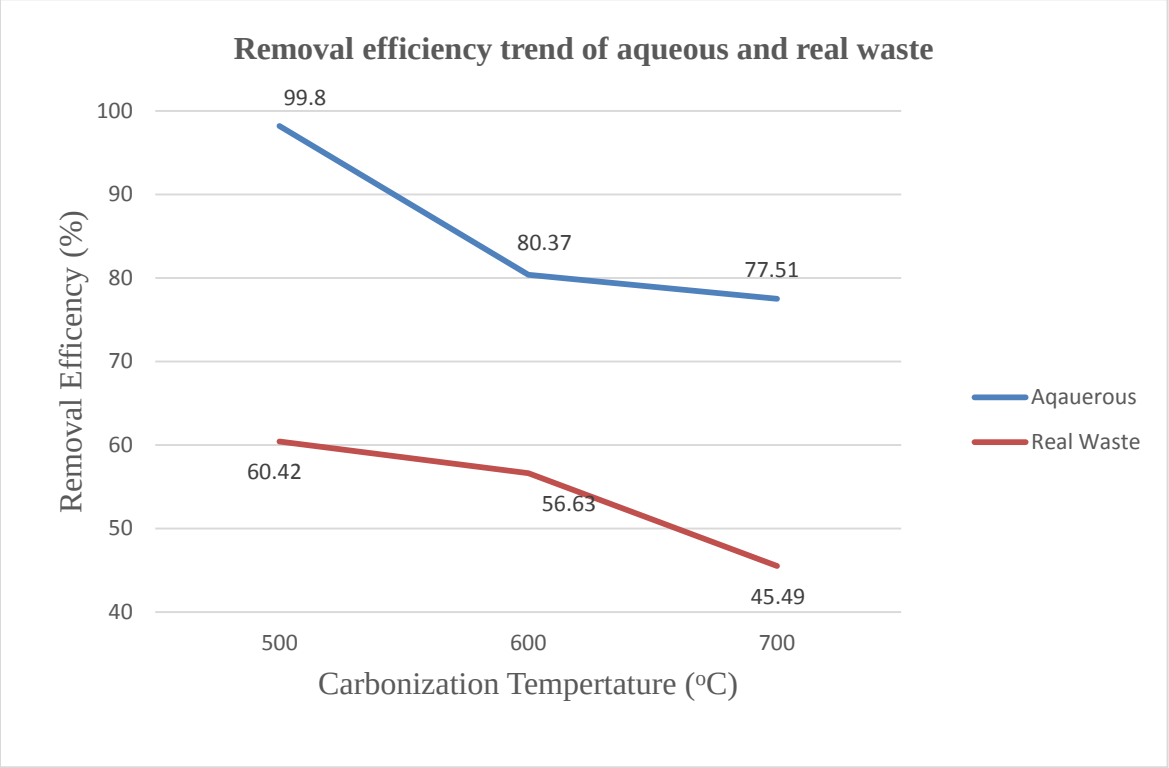


Fig. 4.22 Removal efficiency trend of aqueous and real waste

The performance of 99.8% removal efficiency in aqueous solution (simulated wastewater), declined to 60.42% at 500^0C, 56.63% at 600^0C and 45.49% at 700^0C. The results were much lower than the removal efficiency of synthetic wastewater containing individual metal ions. This can be justified to the fact that the presence other organic solvents, heavy metals ions (cadmium, chromium), binders, additives, BOD5, COD, colors and other toxic material in the solution affected the removal efficiency by competing one another to the adsorbent site.

5. Conclusion

The main conclusions that can be drawn from current investigation are given below. The phyisicochemical characteristics of the bamboo based activated carbon (BAC), were studied. The moisture, yield, volatile matter, the ash, SEM, *XRD*, FTIR and iodine number, the results that has been observed, were highly related with that of previously studied paper.

The adsorption of Pb^{2+} ion from aqueous solution (artificially simulated wastewater) by bamboo activated carbon was investigated. The effect of solution pH, initial metal ion concentration, and adsorbent dose on metal ion removal has been studied. The pH was found the dominant factor and the optimal was found at 5.

.

The performance of 99.8% removal efficiency in simulated wastewater declined to 60.42% at 500^0C, 56.63% at 600^0C and 45.49% at 700^0C The results were much lower than the removal efficiency of synthetic wastewater containing individual metal ions. This can be justified to the fact that the presence other organic solvents, heavy metals (cadmium, chromium), binders, additives, BOD5, COD, colors and other toxic material in the solution affect the removal efficiency by competing one another to the adsorbent site.

Isothermal data of metal ion sorption by bamboo activated carbon indicated that the biosorption process followed both Langmuir and Freundlich models. Data analyzed from models, showed that the Freundlich models adsorption capacity of bamboo activated carbon higher than that of the Langmuir isotherm model.

6. Recommendation

Based on the results gained from the study, the following suggestions have been made further research needs

- For future research, it is recommended Batch kinetic studies at various temperatures; various particle size of BAC and agitation speed may be conducted to determine thermodynamic parameter for lead adsorption on bamboo activated carbon at the optimal condition that has been investigated from the current work.
- Based on the current work, pilot studies should be considered for future research through column studies using industrial wastewater containing lead ions and other metals to investigate effect of competitive adsorption on lead and other metals.
- Some other characteristics of bamboo activated carbon ,useful in wastewater purification, should be investigated, such as for removal of suspended solids, dissolved solids, BOD5,COD, free chlorine, and kjedahl nitrogen(TKN).
- Comparative analysis of bamboo based activated carbon with commercial activated carbon.
- Backwash and recovery of heavy metal from the adsorbent surface for other application.

REFERENCE

Abia, A. A. (2006). A bioseparation process for removing heavy metals from waste water using biosorbents, *African Journal of Biotechnology*, 1167–1179.

Abdel-ghani, N. T., & Elchaghaby, G. A. (2014). Biosorption for metal ions removal from aqueous solutions: a review of resent studies, *Journal of Latest Research in Science and Technology 3*(1), 24–42.

Ahalya, N, Ramachandra, T and Kanamadi, R. (2003). Biosorption of heavy metals. Res. J. Chem. Environ. 7: 71-79.

Ajmal, M., Ali, R., Rao, K., Anwar, S., Ahmad, J., & Ahmad, R. (2003). *Adsorption studies on rice husk: removal and recovery of Cd (II) from wastewater*, Bio resource Technology *86*, 147–149.

Alebel Abebe Belay (2010). Impacts of Chromium from Tannery Effluent and Evaluation of Alternative Treatment Options *Journal of Environmental Protection,* 1**,** 53-58.

Alluri, H. K., Ronda, S. R., Settalluri, V, Suryanarayana, V., & Venkateshwar, P. (2014). recent advances in biosorption of heavy metals: *support tools for biosorption equilibrium, kinetics and mechanism, Acadamia Romana 59*, 527–538.

Amada, S., Y. Ichikawa, T. Munekata, Y. Nagase, and K. Shimizu. (1997). *Fiber texture and mechanical graded structure of bamboo*. Composite Part B. 28(B): 13-20.

Asouhidou D.D., Triantafyllidis K.S., Lazaridis N.K., Matis K.A., Kim S.S., Pinnavaia T.J.,((2009). *Sorption of reactive dyes from aqueous solutions by ordered hexagonal and disordered mesoporous carbons,* Micropor.Mesopor. Mater, 117, 257-267.

Azene Bekele (2007). *Useful trees and shrubs for Ethiopia*. Regional Soil Conservation Unit (RSCU), Technical Handbook No. 5, SID A, Nairobi. 474.

Bonilla-Petriciolet, A. (eds.) *Lignocellulosic precursors used in the synthesis of activated carbon: characterization techniques and applications in the wastewater treatment.* Croatia: In Tech.

Chen W., Parette R., Zou J., Cannon F.S.,Dempsey B.A.(2007), *Arsenic removal by iron-modified activated carbon,* Water Res., 41, 1851-1858.

Chimie, R. R. De. (2014). *Recent advances in biosorption of heavy metals: support tools for biosorption equilibrium, kinetics and mechanism, Acadamia Romana 59*, 527–538.

Demirbas, A. (2008). Heavy metal adsorption onto agro-based waste materials: A review, Journal of Hazardous Materials *157*, 220–229.

Durán-Valle, C. J. (2012). Techniques Employed in the Physicochemical Characterization of Activated Carbons. *In:* MONTOYA, V. H. & PETRICIOLET, A. B. (eds.) *Lignocellulosic Precursors Used in the Synthesis of Activated Carbon -Characterization Techniques and Applications in the Wastewater Treatment.* Croatia: In Tech.

Foo, K. Y. & Hameed, B. H. (2009).Recent *developments in the preparation and regeneration of activated carbons by microwaves*. *Adv. Colloid Interface Sci.*, 149.

Gadd, G. M. (2009). Biosorption: critical review of scientific rationale, environmental importance and significance for pollution treatment, *Published online in Wiley Interscience* (April 2008), 13–28.

Gimba, C., Ocholi, O., & Nok, A. (2004).*Preparation of activated carbon from Agricultural wastes: Kinetic and equilibrium studies*. *Journal of Hazardous Material, **137***, 695-699.

Hameed BH, Din ATM, Ahmad AL, (2006). *Adsorption of Methylene Blue onto Bamboo-Based Activated Carbon: Kinetics and Equilibrium Studies. J. Hazard. Mater*; 141 (3), 819 – 825.

Hernández-Montoya, V., García-Servin, J. & Bueno-López, J. I. (2012). *Thermal Treatments and Activation Procedures Used in the Preparation of Activated Carbons*. *In:* Hernández-Montoya, V. & Bonilla-Petriciolet, A. (eds.) *Lignocellulosic precursors used in the synthesis of activated carbon: characterization techniques and applications in the wastewater treatment.* Croatia: In Tech.

Holant, Z. R. (1995).*Biosorption of Heavy Metals, Biotechnolprog. 11*(235–250).

Igwe, J. C., Abia, A. A., & Ibeh, C. A. (2008). Adsorption kinetics and intraparticulate diffusivities of Hg, As and Pb ions on unmodified and thiolated coconut fiber, *Int. J Environ Sce. 5*(1), 83–92.

Itodo, a U., Oko, O. J., Kamba, E. a, Andrew, C., Bello, H. a, Island, O. B. … State, O. (2014). Adsorptive Removal of Cd^{+2} and Zn^{+2} from Aqueous System by BSG, *Chemistry and Material Research*, *6*(2), 104–112.

Janssen, J.J.A. (1995). *Building with bamboo* (2nd ed.). Intermediate Technology Publication Limited, London. pp. 65.

Jiang J.Q., (2012) Ashekuzzaman S.M., *Development of novel inorganic adsorbent for water treatment*, Curr. Opinion Chem. Eng., 1, 191-199.

Kanamadi, R. D. (2003). Biosorption of Heavy Metals, *Research Journal of Chemistry and Environment, 7*(4), 235–250.

Kasnejad M.H., Esfandiari A., Kaghazchi T., Asasian N., (2012). *Effect of pre-oxidation for introduction of nitrogen containing functional groups into the structure of activated carbons and its influence on Cu (II) adsorption*, J. Taiwan Inst. Chem. Eng., 43, 736-740.

Kassahun Embaye (2003). *Ecological aspects and resource management of bamboo forests in Ethiopia.* Doctoral dissertation, ISSN 1401-6230, Swedish University of Agricultural Sciences, Uppsala, Sweden.

Lakkad, S.C. and J.M. patel. (1980). *Mechanical properties of bamboo, a natural composite*. Fiber Sci. Technol. 14: 319-322.

M. Lawrence and Y. Jiang, (2017). *Porosity, Pore Size Distribution, Micro-structure*: BRE Centre for Innovative Construction Materials, University of Bath.

Masood A, k, Amanual, A, Ramesh D and Abiyu K. (2015). *Removal of lead ion from aqueous solution by bamboo activated carbon. Arba Minch University, Arba Minch, Ethiopia (East Africa).*

Mohamed, A. R., Mohammadi, M. & Darzi, G. N. (2010). Preparation of carbon molecular sieve from lignocellulosic biomass: A review. *Renewable and Sustainable Energy Reviews*, 14**,** 1591-1599.

Okafor, P. C., Okon, P. U., Daniel, E. F., & Ebenso, E. E. (2012). *Adsorption Capacity of Coconut* (Cocosnucifera L.) Shell for Lead, Copper, Cadmium and Arsenic from Aqueous Solutions, Int J. Electrochem Sce. *7*, 12354–12369.

Ojedokun, A. T., & Solomon, O. (2016).*Sequestering heavy metals from wastewater using cow dung. Water Resources and Industry*, *13*, 7–13.

Peláez-cid, A. A. & Teutli-león, M. M. M. (2012). Lignocellulosic Precursors Used in the Elaboration of Activated Carbon. *In:* HERNÁNDEZ-MONTOYA, V. &

Ravikumar, K., & K, P. S. A. (2013). *Heavy Metal Removal from Water using Moringaoleifera Seed Coagulant and Double Filtration*, *IJSR 4*(5), 10–13.

Prakash Kumar BG, Shivakamy K, Miranda LR & Velan M. (2006). Preparation of steam activated carbon from rubber wood sawdust (*Hevea brasiliensis*) and its adsorption kinetics. *Journal of Hazardous Materials* B136: 922–929.

Paulo Fernando Trugilho, Gregório M, Santana (2017). *Development of activated carbon from bamboo (*bambusa vulgaris*) for pesticide removal from aqueous solutions.* Federal University of Lavras.

Sharma, A., & Bhattacharyya, K. G. (2005). Adsorption of Chromium (VI) on AzadirachtaIndica (Neem) Leaf Powder, (VI), *Adsorption* 327–338.

Sulaiman, M. S. (2015). *Factors affecting biosorption of cu (ii) ions from industrial wastewater*, *Applied Research Journal* 311–315.

Tan, I. A. W., Ahmad, A. L. & Hameed, B. H. (2007). Adsorption of Basic Dye using Activated Carbon Prepared from Oil Palm Shell: Batch and Fixed Bed Studies. *Desalination,* 225**,** 13-28.

Tsezos, M., Remoundaki, E., & Hatzikioseyian, A. biosorption - principles and applications for metal immobilization from waste-water Streams, *Workshop on Cleaner Production,* 30–40.

Tovar-Gomez R., Rivera-Ramirez D.A., Hernandez-Montoya V., Bonilla-Petriciolet A., Duran-Valle C.J., Montes-Moran M.A. (2012). *Synergic adsorption in the simultaneous removal of acid blue 25 and heavy metals from water using a Ca(PO 3) $^{2-}$modified carbon*, J. Hazard. Mater, 199-200, 290-300.

Venkateswarlu, P., Ratnam, M. V., Rao, D. S., & Rao, M. V. (2007). *Removal of chromium from an aqueous solution using Azadirachtaindica (neem) leaf powder as an adsorbent*, *International Journal of Physical Science 2*(August), 188–195.

Volesky, B. (2001). Detoxification of metal-bearing effluents: biosorption for the next century *Hydrometalurgy*, 59(2001), 203-216

Wang, J., & Chen, C. (2009).Biosorbents for heavy metals removal and their future. *Biotechnology Advances*, *27*(2), 195–226.

Wasewar KL, Mohammad A, Prasad B and Mishra IM. (2008a) Adsorption of Zn using factory tea waste: kinetics, equilibrium and thermodynamics. CLEAN: Soil, Water, Air; 36(3): 320-329.

www.wikipedia.com

Xiaojum. Ma,(2014).*Preparation, Surface and Pore Structure of high Surface area Activated Carbon fibers from Bamboo by stem Activation*. Tianjin University of Science & Technology, Tianjin 300222, China.

YE, C.Y. ET AL. 1989.*Integration utilization of bamboo resource*, Shanghai Science and Technology Press, Shanghai, China.

Yu, B., Zhang, Y., Shukla, A., Shukla, S. S., & Dorris, K. L. (2001).*The removal of heavy metals from aqueous solutions by sawdust adsorption removal of lead and comparison of its adsorption with copper*, Journal of Hazardous Material *84*, 83–94.

Zabochnicka-świątek, M., & Krzywonos, M. (2014). Potentials of Biosorption and Bioaccumulation Processes for Heavy Metal Removal, *Pol J Environ. Stud. 23*(2), 551–561.

Zhou Fangchun. 1998. *Bamboo forest cultivation. China Forestry Publishing* House, Beijing, China.

Appendix A

Specific *gravity of bamboo based activated carbon*

$$G_s = \frac{M_2 - M_1}{(M_2 + M_4) - (M_1 + M_3)}$$

At 500^{0}C *M_2= mass of AC inside pycnometer = 138.2gm, M_1= mass of pycnometer =108.5gm*

M_3= mass of oven dried AC inside the pycnometer filled with water =366.3gm

M_4= mass of pycnometer filled with water =356.7gm

$G_s@500 = \frac{138.2-108.5}{(138.2+356.7)-(108.5+366.3)}$= 1.470

At 700^{0}C *M_1= mass of pycnometer =108.5gm, M_2= mass of AC inside pycnometer=128.5gm*

M_3= mass of oven dried AC inside the pycnometer filled with water=363.8gm

M_4= mass of pycnometer filled with water=356.7gm

$$G_s@700 = \frac{128.5 - 108.5}{(128.5 + 356.7) - (108.5 + 363.8)} = 1.550$$

Porosity Estimation

$$n = 1 - \frac{\rho}{\rho_w G_{s(1+w)}}$$

At 500^{0}C the porosity were calculated

*n= 1−0.475/1gm/cm^3*1.470 (1+0.077)*

<u>n=0.70422</u>

At 600^{0}C the porosity were calculated

*n= 1−0.475/1gm/cm^3*1.486 (1+0.071)*

<u>n=0. 6962</u>

At 700^{0}C porosity were calculated

*n= 1−0.4901/1gm/cm^3*1.550 (1+0.0602)*

<u>n=0.6926</u>

Carbon Yield

Each 100gm of chopped bamboo raw was used for carbonization after 3 hours the carbon yield were estimated at 500, 600 and 700^{0}C

At 500^{0}C of carbonization

$$\mathrm{Y_{ch}} = \frac{\mathrm{w_{ch}}}{\mathrm{w_0}} \times 100\%$$

$\mathrm{w_o} = 100\mathrm{gm}.\ \mathrm{w_{ch}} = 40.6\mathrm{gm}$

$$Y_{ch} = \frac{40.6}{100} \times 100\%$$

$$\underline{Y_{ch} = 40.6\ At\ 500^0\mathrm{C}}$$

At 600^{0}C of carbonization

$$w_o = 100gm w_{ch} = 38.04gm$$

$$Y_{ch} = \frac{w_{ch}}{w_0} \times 100\%$$

$$\frac{38.04}{100} \times 100\%$$

$$\underline{Y_{ch} = 38.04\ at\ 600^0\mathrm{C}}$$

At 700^{0}C of carbonization

$$w_o = 100gm w_{ch} = 36.01gm$$

$$Y_{ch} = \frac{36.01}{100} \times 100\%$$

$$\underline{Y_{ch} = 36.01\ at\ 700^0\mathrm{C}}$$

Moisture content

$$w = \frac{w_0 - w_d}{w_0} \times 100\%$$

At 500^{0}C w_0= 1gm w_d= 0.923

$$w = \frac{1gm - 0.923}{1gm} \times 100\%$$

$$w = \underline{0.077 \text{ or } 7.7\%}$$

At 600^{0}C w_0= 1gm w_d= 0.9299gm

$$w = \frac{1gm - 0.9299gm}{1gm} \times 100\%$$

w =0.0701 or 7.01%

At 700^0C w_0= 1gm w_d= 0.938gm

$$w = \frac{1gm - 0.938gm}{1gm} \times 100\%$$

w =0.0620 or 6.20%

Ash content

$$\text{Ash cont.} = \frac{W_{ash}}{W_o} \times 100\%$$

At 500^0C of carbonization

$$\frac{0.0351\text{gm}}{1\text{gm}} \times 100\%$$

3.51%

At 600^0C of carbonization

$$\frac{0.0473\text{gm}}{1\text{gm}} \times 100\%$$

4.73%

At 700^0C of carbonization

$$\frac{0.0553\text{gm}}{1\text{gm}} \times 100\%$$

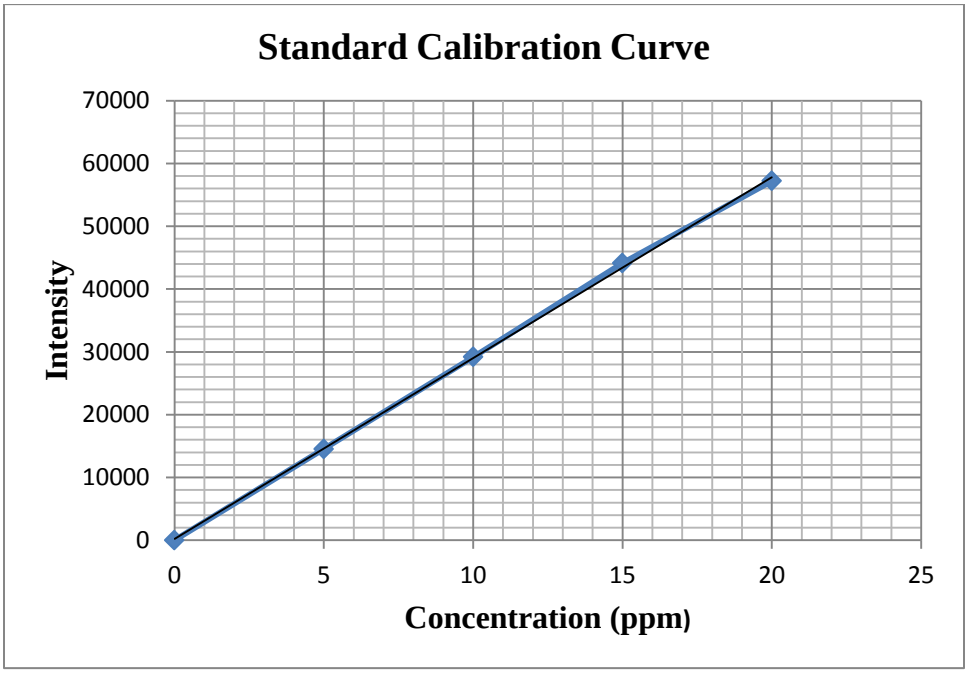
5.53%

Appendix B

Metal: Pb

Detection limit = 0.0001ppm

Standards	Conc. (ppm)	Intensity
Blank	0	0.0000
Standard 1	5	14543.7483
Standard 2	10	29172.9983
Standard 3	15	44130.2424
Standard 4	20	57204.3578
Standard 5	25	72161.601
Standard 6	30	85235.71



Sample	Conc. (ppm)
Blank	0.1719
E1	12.3472
E2	9.2097
E3	4.5980
E4	11.1678
E5	9.9120
E6	8.0332
E7	15.9384
E8	8.3914
E9	12.6068
E10	1.0180
E11	0.4717
E12	1.0719
E13	1.6241
E14	2.0032
E15	1.7581
E16	1.4720
E17	1.2606
E18	12052
E19	12.1690
E20	13.9298
E21	14.8136
E22	11.9986
E23	12.2097
E24	10.6105
E25	25.9245
E26	21.9590

E27	19.8568
E28	19.6211
E29	22.4247
E30	11.8716
E31	13.0091
E32	16.3519

Run	Initial Concentration (mg/l)	Final concentration (mg/l)	Average Adsorption amount (mg/g)	Dose(gm.)	P^H	Percent removal
E1	50	12.3472	9.4132	1	6	75.30
E2	50	9.2097	6.798	1.5	6	81.58
E3	50	4.5980	5.675	2	6	90.80
E4	100	11.1678	22.20	1	6	88.83
E5	100	9.9120	15.01	1.5	6	89.08
E6	100	8.0332	11.49	2	6	88.96
E7	150	15.9384	33.51	1	6	89.70
E8	150	14.1914	22.63	1.5	6	90.2
E9	150	12.6068	17.17	2	6	91.5
E10	50	1.0180	12.24	1	5	97.9
E11	50	0.4717	8.25	1.5	5	99.0
E12	50	1.0719	6.116	2	5	99.8
E13	100	1.6241	24.59	1	5	98.3
E14	100	2.0032	16.32	1.5	5	97.9
E15	100	1.7581	12.28	2	5	98.2
E16	150	1.4720	37.13	1	5	99.0
E17	150	1.2606	24.78	1.5	5	99.1
E18	150	1.2052	18.59	2	5	94.2
E19	50	12.1690	9.457	1	3	75.6
E20	50	13.9298	6.011	1.5	3	72.1
E21	50	14.8136	4.398	2	3	70.37
E22	100	11.9986	22.0	1	3	87.0
E23	100	12.2097	14.63	1.5	3	88.7
E24	100	10.6105	11.17	2	3	89.3
E25	150	25.9245	31.04	1	3	82.0

E26	150	21.9590	21.34	1.5	3	85.3
E27	150	19.8568	16.26	2	3	86.7
E28	100@600^{0}C	19.6211		1.5	5	80.37
E29	100@700^{0}C	22.4247		1.5	5	77.57
E30	30@500^{0}C	11.8716		2	5	60.42
E31	30@600^{0}C	13.0091		2	5	56.63
E32	30@700^{0}C	16.3519		2	5	45.49

Design expert 6.0.8 response

Std	Run	Block	Factor 1 A: pH	Factor 2 B:concentratio mg/l	Factor 3 C:dose gm	Response 1 Response 1 %
5	1	Block 1	5.00	100.00	1.00	98.3
4	2	Block 1	3.00	100.00	1.00	87
14	3	Block 1	5.00	100.00	1.50	97.9
20	4	Block 1	5.00	50.00	2.00	99.8
29	5	Block 1	5.00	100.00	1.50	97.9
9	6	Block 1	6.00	150.00	1.00	89.7
21	7	Block 1	6.00	50.00	2.00	90.8
22	8	Block 1	3.00	100.00	2.00	89
7	9	Block 1	3.00	150.00	1.00	82
16	10	Block 1	3.00	150.00	1.50	85.3
2	11	Block 1	5.00	50.00	1.00	97.9
1	12	Block 1	3.00	50.00	1.00	75.6
24	13	Block 1	6.00	100.00	2.00	88.96
3	14	Block 1	6.00	50.00	1.00	75.3
11	15	Block 1	5.00	50.00	1.50	99
25	16	Block 1	3.00	150.00	2.00	86.7
28	17	Block 1	5.00	100.00	1.50	97.9
18	18	Block 1	6.00	150.00	1.50	90.2
17	19	Block 1	5.00	150.00	1.50	99.1
13	20	Block 1	3.00	100.00	1.50	88.7
27	21	Block 1	6.00	150.00	2.00	91.5
15	22	Block 1	6.00	100.00	1.50	89.08
10	23	Block 1	3.00	50.00	1.50	72.1
23	24	Block 1	5.00	100.00	2.00	98.2
32	25	Block 1	5.00	100.00	1.50	97.9
30	26	Block 1	5.00	100.00	1.50	97.9

Appendix C

IR-Spectroscopy: Functional Group Identification

Infrared spectroscopy (IR spectroscopy or Vibrational Spectroscopy) is the spectroscopy that deals with the infrared region of the electromagnetic spectrum that is light with a longer wavelength and lower frequency than visible light. It covers a range of techniques, mostly based on absorption spectroscopy (www.wikipedia.com). The exact frequency at which a given vibration occurs is determined by the strengths of the bonds involved and the mass of the component atoms. For a more detailed discussion of these factors. In practice, infrared spectra do not normally display separate absorption signals for each of the 3n-6 fundamental vibrational modes of a molecule.

The number of observed absorptions may be increased by additive and subtractive interactions leading to combination tones and overtones of the fundamental vibrations, in much the same way that sound vibrations from a musical instrument interact. Furthermore, the number of observed absorptions may be decreased by molecular symmetry, spectrometer limitations, and spectroscopic selection rules. One selection rule that influences the intensity of infrared absorptions is that a change in dipole moment should occur for a vibration to absorb infrared energy. Absorption bands associated with C=O bond stretching are usually very strong because a large change in the dipole takes place in that mode.

Some General Trends

- Stretching frequencies are higher than corresponding bending frequencies. (It is easier to bend a bond than to stretch or compress it).
- Bonds to hydrogen have higher stretching frequencies than those to heavier atoms.
- Triple bonds have higher stretching frequencies than corresponding double bonds, which in turn higher frequencies than single bonds. (Except for bonds to hydrogen).

Functional Group Name	Adsorption Range (cm^{-1})	Types of Vibration causing IR absorption
Alkanes	3000-2800	H-C-H Asymmetric & symmetric stretch
	1500-1440	H-C-H Bend
Alkenes	3100-3000	C=C-H Asymmetric stretch
	1675-1600	C-C=C Symmetric stretch
Alkynes	3300-3200	=C-H stretch
	2200-2100	C-C stretch
Aromatic Rings	3100-3000	C=C-H Asymmetric stretch
	1600-1580	C-C=C Symmetric stretch
	1500-1450	C-C=C Asymmetric stretch
Phenol & Alcohols	3600-3100	Hydrogen-bonded O-H stretch
	1730-1650	C=O stretch
Ketones	1750-1625	C=O stretch
	1750-1625	C=O stretch
Aldehydes	2850-2800	C-H stretch off C=O
Esters	1755-1650	C=O stretch
	1300-1000	C-O stretch
Ether	(1300-1000)	(C-O stretch)
Amines- Primary	3500-3100 (two peaks)	N-H stretch
	1640-1560	N-H bend

Amines – Secondary	3500-3100 (one peak)	N-H stretch
	1550-1450	N-H bend
Nitriles	2300-2200	C-N stretch
Nitro Groups	1600-1500	N=O stretch
	1400-1300	N=O bend
Amides	3500-3100	N-H (stretch similar to amines)
	1670-1600	C=O stretch
	1640-1550	N-H bend
Aldehydes	2750-2700	C-H stretch off C=O

Appendix D

Pictures throughout the experiments

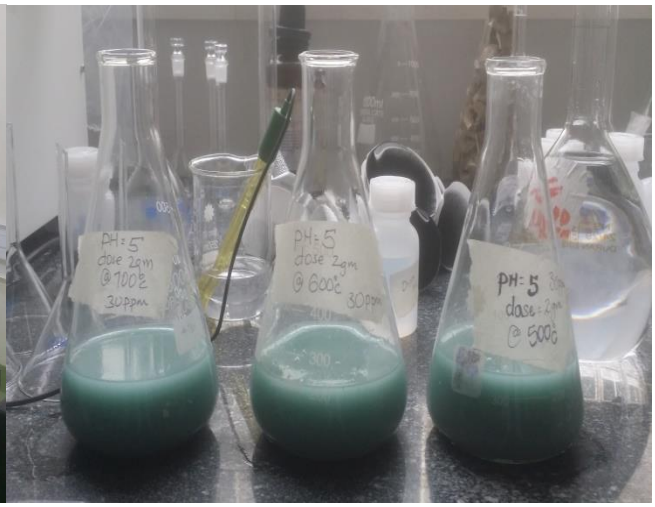
Solution preparation

orbital shaker

filteration of the solution

digestion process

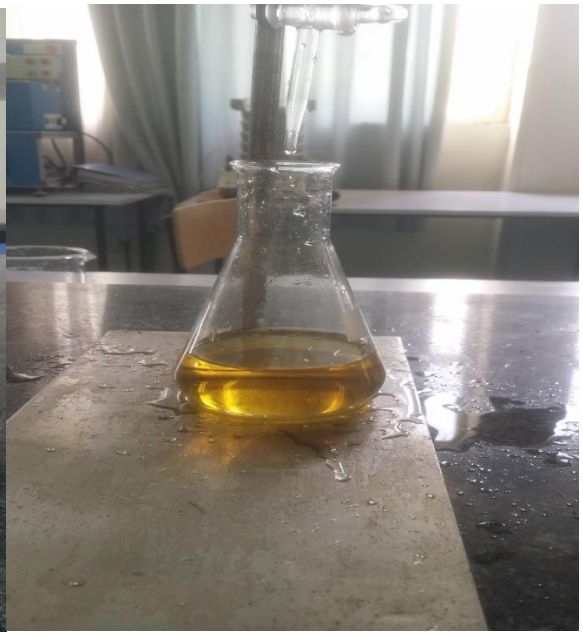
SEM

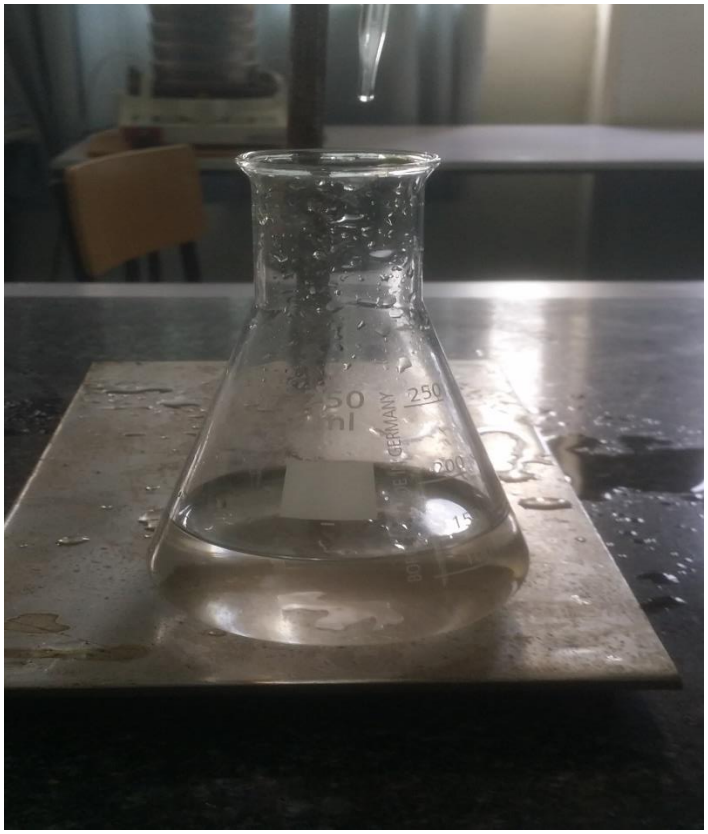


Real waste

Filter one

pycnometer (specific gravity)





Iodine number determination

Lab seasion

filtered sample for AAS